doi:10.1038/nature17151

Synthesis of monomers

A. General information
B. Synthetic route to monomer **1a**
C. Synthetic route to monomer **1b**

A. General information

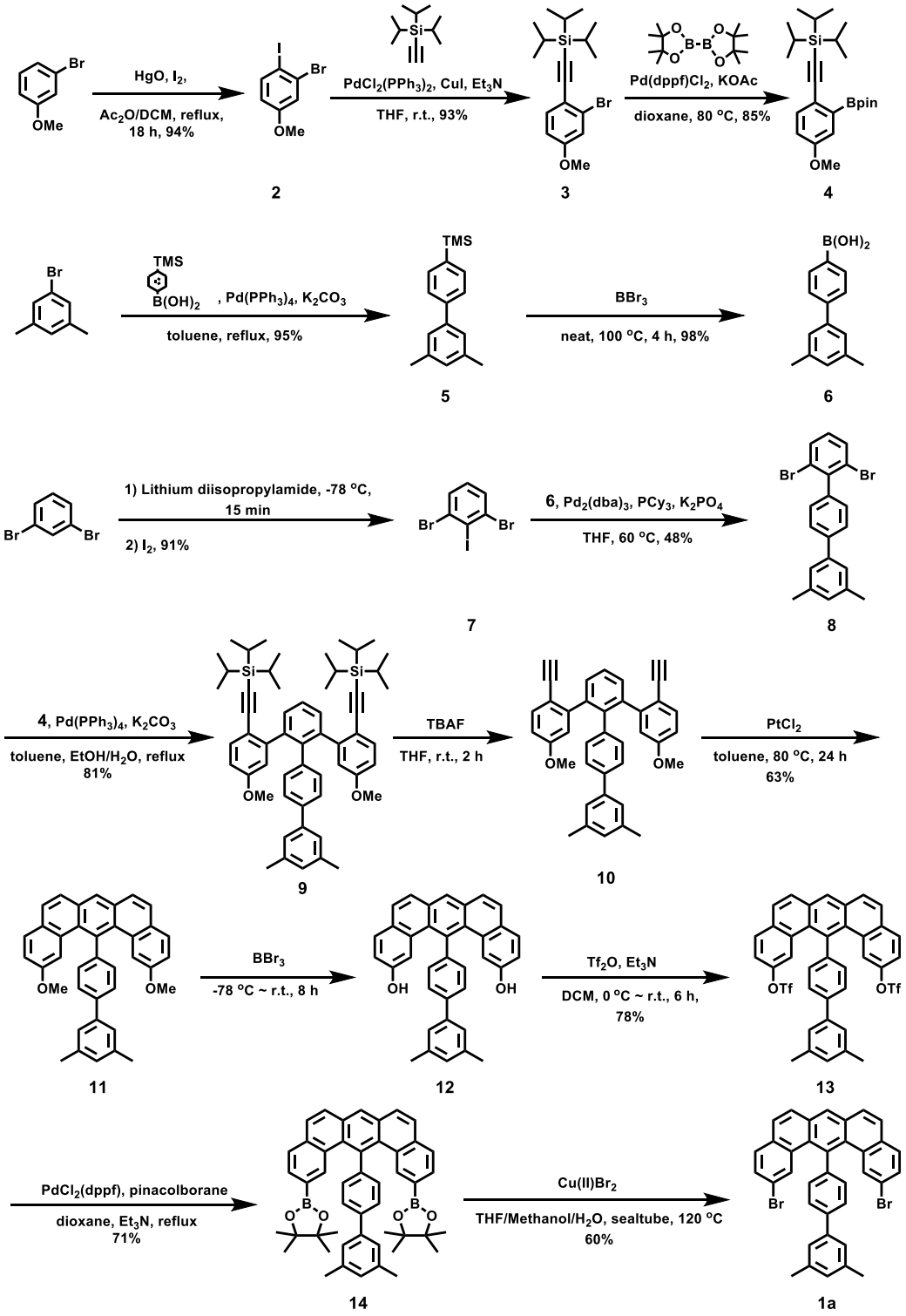
All reactions dealing with air- or moisture-sensitive compounds have been carried out in the dry reaction vessel under argon protection. Preparative column chromatography has been performed on silica gel from Merck with a grain size of 0.063-0.200 mm (silica gel). Melting points have been determined on a Büchi hot stage apparatus and were uncorrected. NMR spectra have been measured on Bruker DPX 250, AMX 300, and DRX500 spectrometers, and referenced to residual signals of the deuterated solvent. Abbreviations: s = singlet, d = doublet, dd = double doublet, t = triplet, m = multiplet. The high-resolution electrospray ionization mass spectrometry (HR-ESI-MS) has been performed on an ESI-Q-TOF system (maXis, BrukerDaltonics, Germany), where the instrument is operated in wide pass quadrupole mode for MS experiments, with the TOF data being collected between m/z 100–5000. The high-resolution time-of-flight mass spectrometry (APPI-TOF and MALDI-TOF) measurements have been performed on a SYNAPT G2 Si high resolution time-of-flight mass spectrometer (Waters Corp., Manchester, UK) with matrix-assisted laser desorption/ionization (MALDI) or atmospheric pressure photoionization (APPI) source. For MALDI-TOF MS measurement, the samples have been mixed with DCTB ({(2E)-2-methyl-3-[4-(2-methyl-2-propanyl)phenyl]-2-propen-1-ylidene}malononitrile) and dropped on a MALDI sample plate. For APPI TOF MS measurement, the samples have been diluted in toluene to 5 ppm and then infused into the ionization source directly by a Legato 185 syringe pump (KD Scientific, MA, USA) at a flow rate of 5 μL/min. The mass spectrometer has been calibrated against red phosphors under MALDI mode previously and the spectra have been recorded using C60 as lockmass.

Unless otherwise noted, materials have been purchased from Fluka, Aldrich, Acros, ABCR, Merck and other commercial suppliers and used as received without further purification.

B. Synthetic route to monomer **1a**

"U-shaped" monomer **1a** was synthesized following the synthetic route described in supplementary Scheme 1. As shown in the Scheme 1, compound **2** was obtained after iodination of 1-bromo-3-methoxybenzene, which had been reported previously [1]. Then, after Sonogashira coupling reaction with (triisopropylsilyl)acetylene, compound **3** was obtained in 93% yield. Afterwards, compound **4** was synthesized after borylation of **3** with bis(pinacolato)diboron in 85% yield[2]. In parallel, compound **5** was prepared by Suzuki coupling of 1,3-dimethyl-5-bromobenzene with 4-(trimethylsilyl) phenylboronic acid in 95% yield. Then, compound **5** was treated with boron tribromide (BBr_3) under neat condition to afford compound **6** in 98% yield [3]. Meanwhile, compound **7** was synthesized *via* an iodination [4] of 1, 3-dibromobenzene with iodine in 91% yield. Then, compound **8** was obtained after a selective Suzuki coupling of **7** with **6** in 48% yield [5].

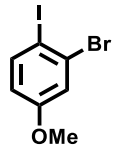
In the following steps, compound **9** was prepared after a two-fold Suzuki coupling of **4** with **8** in 48% yield and subsequently treated with tetrabutylammonium fluoride (TBAF) solution for the de-protection reaction to remove triisopropyl (Tips) group [6] to afford compound **10** in 99% yield. The crude compound **10** was used directly for the next step. Compound **11** was then prepared after a catalytic cyclization by treating **10** with platinum (II) chloride catalyst in 63% yield [7]. Following by a step of demethylation [8] by treating **11** with BBr_3, crude compound **12** was obtained and used directly for the next step. Afterwards, compound **13** was yielded by treating **12** with trifluoromethanesulfonic anhydride (Tf_2O) in 78% yield [9]. Later on, compound **14** was synthesized after borylation of **13** with pinacolborane in 71% yield [10]. At last, target monomer **1a** was obtained in 60% yield after treatment of **14** with copper (II) bromide in a seal tube at 120 °C [10,11].



Supplementary Scheme 1. Synthetic route towards monomer **1a.**

Experimental details and description

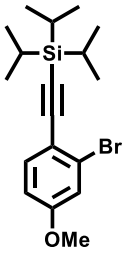
3-Bromo-4-iodoanisole (2)



A stirred solution of 3-bromoanisole (10 g, 53.5 mmol), mercury (II) oxide (8.8 g, 40.6 mmol), and acetic anhydride (1 mL) in dichloromethane (100 mL) was refluxed for 30 min. Then, the iodine (17.6 g, 69.5 mmol) was added by six portions every 30 min. After refluxing for 12 h and filtration over a pad of celite, the filtrate was washed with a saturated sodium thiosulfate solution. The aqueous layer was extracted with dichloromethane (3 × 10 mL), the combined organic layers were dried over sodium sulfate, and evaporated to remove solvent. Then, purification by flash chromatography (eluent: cyclohexane) afforded the titled compound **2**.

Colorless oil (Yield = 94%). ^{1}H NMR (300 MHz, CD_2Cl_2) δ 3.77 (s, 3H), 6.62 (dd, J = 8.8, 2.9 Hz, 1H), 7.21 (d, J = 2.9 Hz, 1H), 7.71 (d, J = 8.8 Hz, 1H); ^{13}C NMR (75 MHz, CD_2Cl_2) δ 56.19, 89.80, 115.84, 118.96, 130.31, 140.79, 160.85.

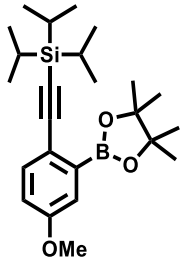
((2-Bromo-4-methoxyphenyl)ethynyl)triisopropylsilane (3)



To a mixture of aryl iodide compound **2** (10 g, 32 mmol), bis-(triphenylphosphine)palladium (II) dichloride (448 mg, 0.64 mmol), copper(I) iodide (243.4 mg, 1.28 mmol), triethylamine (14 mL) in tetrahydrofuran (100 mL) was added drop wise under an argon atmosphere. Next, the liquid compound (triisopropylsilyl) acetylene (8.74 g, 48 mmol) was added *via* a syringe as well. After the mixture was stirred at room temperature overnight, diethyl ether (20 mL) was added to the crude mixture. Then the mixture was filtered over a short pad of celite to remove catalyst. The organic layer was washed with brine (5 mL) for three times, and the organic layer was collected and dried over magnesium sulfate and evaporated. Purification by flash chromatography (eluent: 1% diethyl ether/hexane) afforded the compound **3**.

Colorless oil (Yield = 93%); ^{1}H NMR (300 MHz, CD_2Cl_2) δ 1.16 (s, 21H), 3.80 (s, 3H), 6.82 (dd, J = 8.7, 2.6 Hz, 1H), 7.14 (d, J = 2.5 Hz, 1H), 7.44 (d, J = 8.6 Hz, 1H); ^{13}C NMR (75 MHz, CD_2Cl_2) δ11.98, 19.07, 56.24, 94.56, 105.40, 113.98, 118.33, 118.37, 126.89, 135.07, 160.61; HRMS (MALDI-TOF, positive) m/z calcd for $C_{18}H_{27}BrOSi[M]^+$ 366.1015, found 366.1078.

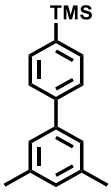
Triisopropyl((4-methoxy-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)ethynyl)silane (4)



A 250 mL round flask was charged with compound **3** (19.5 g, 53.1 mmol), bis(pinacolato)diboron (14.8 g, 58.4 mmol), potassium acetate (15.6 g, 159 mmol) and [1,1'-bis(diphenylphosphino)ferrocene]dichloropalladium(II) (1.2 g, 1.6 mmol). Then the stirring mixture was degassed by argon bubbling for 20 min. Afterwards, the mixture was stirred overnight at 80 °C under an argon atmosphere. After cooling to room temperature, the mixture was washed with water and extracted with ethyl acetate (20 mL × 3). The combined organic layer was washed with brine, dried over magnesium sulfate, and evaporated. At last, the crude residue **4** was purified by passing through a shot pad of silica gel (eluent: 10% ethyl acetate/hexane) to remove the catalyst and used directly for the next step.

Brown yellow oil (Yield = 85%). ^{1}H NMR (300 MHz, CD_2Cl_2) δ 1.15 (d, J = 1.2 Hz, 23H), 1.33 (s, 12H), 3.81 (s, 3H), 6.89 (dd, J = 8.6, 2.9 Hz, 1H), 7.23 (d, J = 2.8 Hz, 1H), 7.45 (d, J = 8.5 Hz, 1H); ^{13}C NMR (75 MHz, CD_2Cl_2) δ11.86, 18.97, 25.00, 55.63, 84.28, 90.90, 108.06, 116.72, 120.34, 120.80, 135.85, 159.17; HRMS (ESI, positive) m/z calcd for $C_{24}H_{40}BO_3Si[M+H]^+$ 415.2840, found 415.2845.

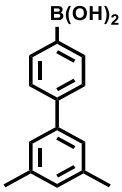
(3', 5'-Dimethyl-[1,1'-biphenyl]-4-yl)trimethylsilane (5)



A 250 mL round flask was charged with 1-bromo-3,5-dimethylbenzene (6 g, 32.4 mmol), (4-(trimethylsilyl)phenyl)boronic acid (9.44 g, 48.6 mmol), potassium carbonate solution (18 g in 10 mL water), ethanol 10 mL, and toluene 50 mL. The mixture was degassed by argon bubbling for 10 min. Then tetrakis(triphenylphosphino)palladium(0) (1.87 g, 1.62 mmol) was added. The resulting mixture was further degassed by argon bubbling for 10 min, and treated with liquid nitrogen bath. After three times freeze-pump-thaw procedure, the mixture was refluxed overnight. The reaction was monitored by thin-layer chromatography plate. Once the reaction was completed, the mixture was washed with deionized water and the aqueous layer was extracted with ethyl acetate for three times (10 mL × 3). The combined organic layer was washed with brine, dried over sodium sulfate, and evaporated. The crude product was purified by silica gel column chromatography (eluent: 5% dichloromethane/hexane) to afford compound **5**.

Colorless oil (Yield = 95%). ^{1}H NMR (300 MHz, CD_2Cl_2) δ 0.32 (s, 9H), 2.39 (s, 6H), 7.02 (m, 1H), 7.24 (ddd, J = 1.9, 1.3, 0.7 Hz, 2H), 7.60 (t, J = 1.3 Hz, 4H); ^{13}C NMR (75 MHz, CD_2Cl_2) δ -0.83, 21.72, 125.47, 126.87, 129.55, 134.34, 138.90, 139.65, 141.48, 142.27.

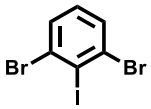
(3',5'-dimethyl-[1,1'-biphenyl]-4-yl)boronic acid (6)



Compound **5** (8 g, 31.4 mmol) was directly treated with neat boron tribromide (12.6 g, 50.3mmol) under argon atmosphere. A condenser charged with argon was attached, and the mixture was heated to 100 °C for 4 h. Once cooled, excess boron tribromide was distilled off under vacuum at room temperature. The resulting gray-purple solid was dissolved in dry hexane (50 mL) and cooled to 0 °C with an ice bath. Water was slowly added drop wise while stirring vigorously until the reaction had been fully quenched. The resulting mixture was filtered and the white solid was washed with deionized water and hexane. The white powder was dried at 80 °C under vacuum overnight, yielded boronic acid **6**, which was used directly for the next step.

White powder.

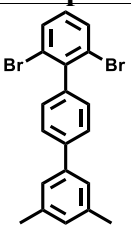
1,3-Dibromo-2-iodobenzene (7)



At -75 °C, butyllithium (42.4 mmol) in hexane (50 mL) and diisopropylamine (42.4 mmol) were added successively to tetrahydrofuran (20 mL). After 15 min, 1, 3-dibromobenzene (5.12 mL, 10 g, 42.4 mmol) was added. The mixture was kept at -75 °C for 2 h before a solution of iodine (10.76 g, 42.4 mmol) in tetrahydrofuran (50 mL) was added. After addition of a 10% aqueous solution (0.10 L) of sodium thiosulfate, the mixture was extracted with diethyl ether for three times (10 mL × 3). The combined organic layer was washed with water and brine once, and then dried over sodium sulfate before being evaporated to dryness. Upon crystallization from ethanol (100 mL), the colorless platelets were obtained.

Colorless platelets (Yield = 91%). ^{1}H NMR (300 MHz, CD_2Cl_2) δ 7.10 (t, J = 8.0 Hz, 1H), 7.58 (d, J = 8.1 Hz, 2H); ^{13}C NMR (75 MHz, CD_2Cl_2) δ109.67, 131.09, 131.73.

2,6-Dibromo-3'',5''-dimethyl-1,1':4',1''-terphenyl (8)

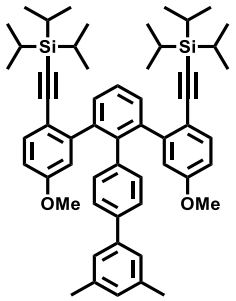


In a glove box, tris(dibenzylideneacetone)dipalladium(0) (1.02 g, 1.11 mmol), tricyclohexylphosphine (1.25 g, 4.45 mmol), compound **7** (8.04 g, 22.23 mmol), and boronic acid **6** (5.03 g, 22.23 mmol) were added to a reaction vessel that was equipped with a stir bar. The degassed tripotassium phosphate (14.16 g, 66.7 mmol) water solution was then added, followed by 100 mL anhydrous tetrahydrofuran. The reaction mixture was then stirred at 60 °C for 3 days. Once the reaction was finished, the reaction mixture was diluted with ethyl acetate, and then extracted by ethyl acetate for three times. The combined organic layer was washed three times with water and once with brine, then

dried over magnesium sulfate and evaporated. The final product **8** was obtained after purification by silica gel column chromatography (eluent: 10% dichloromethane/hexane).

Colorless oil (Yield = 48%). Mp: 104.2-104.9 °C; ^{1}H NMR (300 MHz, CD_2Cl_2) δ 2.40 (s, 6H), 7.04 (tt, J = 1.6, 0.8 Hz, 1H), 7.12 (t, J = 8.0 Hz, 1H), 7.23 – 7.35 (m, 4H), 7.64 – 7.73 (m, 4H); ^{13}C NMR (75 MHz, CD_2Cl_2) δ 21.72, 125.08, 125.52, 127.31, 129.70, 130.19, 130.56, 132.50, 138.96, 140.58, 140.89, 141.62, 143.25; HRMS (APPI-TOF, positive) m/z calcd for $C_{20}H_{16}Br_2$ $[M]^+$ 413.9619, found 413.9619.

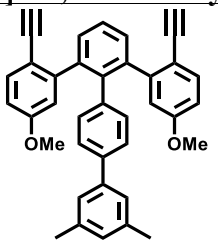
2,6-di[5-methoxy-2-((triisopropylsilyl)ethynyl)phenyl]-3'',5''-dimethyl-1,1':4',1''-terphenyl (9)



A 100 mL round flask was filled with compound **8** (1.16 g, 2.79 mmol), boronic ester **4** (3.47 g, 8.36 mmol) in 50 ml toluene and 5 mL potassium carbonate (2.31 g, 16.72 mmol) water solution. After degassed by argon bubbling for 10 min, the tetrakis(triphenylphosphino)palladium(0) (322 mg, 0.28 mmol) was added. The reaction mixture was then refluxed overnight, and stopped after thin-layer chromatography indicated that the starting material was totally converted. After cooling down to room temperature, the mixture was extracted with ethyl acetate for three times (10 mL x 3), and then the combined organic layer was washed three times with water and dried over magnesium sulfate, then evaporated. The residue was purified by silica gel column chromatography (eluent: 10% ethyl acetate/hexane) yielded compound **9**.

Yellow solid (Yield = 81%). Mp: 180.8-181.2 °C; ^{1}H NMR (300 MHz, CD_2Cl_2) δ 1.02 (s, 42H), 2.30 (s, 6H), 3.50 (s, 6H), 6.30 (d, J = 2.6 Hz, 2H), 6.63 (dd, J = 8.6, 2.7 Hz, 2H), 7.05 – 7.17 (m, 4H), 7.17 – 7.25 (m, 2H), 7.38 (q, J = 1.9 Hz, 4H), 7.42 (s, 1H); ^{13}C NMR (75 MHz, CD_2Cl_2) δ 11.92, 19.04, 21.64, 55.64, 92.66, 107.35, 113.62, 115.91, 116.48, 125.05, 125.93, 126.87, 129.25, 130.72, 131.26, 133.81, 138.72, 139.11, 139.42, 139.46, 140.87, 141.09, 147.54, 159.20; HRMS (ESI, positive) m/z calcd for $C_{56}H_{71}O_2Si_2[M+H]^+$ 831.4993, found 831.4979.

2,6-di[5-methoxy-2-ethynyl-phenyl]-3'',5''-dimethyl-1,1':4',1''-terphenyl (10)

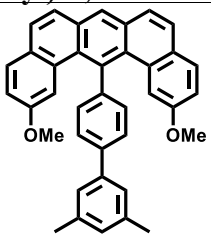


To a solution of compound **9** (1.5 g, 1.8 mmol) in 50 mL of dry tetrahydrofuran, a solution of tetra-*n*-butylammonium fluoride (5.69 g, 18 mmol) in tetrahydrofuran (10 mL) was added. After stirring at room temperature for 2 h, water was added to the reaction mixture and tetrahydrofuran was removed in *vacuo* at 40 °C. The resulting

suspension was extracted three times with ethyl acetate, and the combined organic layer was washed five times with water, dried over magnesium sulfate, and evaporated. The yielded white solid was used directly for the next step without further purification.

White solid.

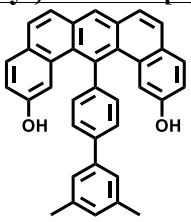
14-(3',5'-dimethyl-[1,1'-biphenyl]-4-yl)-2,12-dimethoxy-dibenzo[a,j]anthracene (11)



A 100 mL round flask was charged with compound **10** (1.1 g, 2.12 mmol) and platinum (II) chloride (56.4 mg, 0.21 mmol). Then the mixture was kept under vacuum conditions for 20 min and refilled with argon. 60 ml of anhydrous toluene was added by syringe afterwards. The mixturewas heated at 80 °C for 24 h after reaction was completed, as judged by thin-layer chromatography (TLC). The toluene was then removed under vacuum conditions and the residue was purified by silica gel column chromatography (eluent: 5% dichloromethane/hexane) yielded the compound **11**.

White solid (Yield = 63%). Mp: 234.4-235.2 °C; ^{1}H NMR (300 MHz, CD_2Cl_2) δ 2.44 (s, 6H), 3.27 (s, 6H), 7.00 (s, 3H), 7.04 (d, J = 2.5 Hz, 1H), 7.10 (s, 1H), 7.31 (dt, J = 1.5, 0.8 Hz, 2H), 7.61 – 7.77 (m, 8H), 7.87 – 7.95 (m, 2H), 8.36 (s, 1H); ^{13}C NMR (75 MHz, CD_2Cl_2) δ 21.77, 55.13, 111.74, 117.12, 125.18, 125.37, 128.33, 129.18, 129.92, 130.09, 130.23, 132.45, 132.86, 133.15, 139.34, 144.96, 156.99 ; HRMS (ESI, positive) m/z calcd for $C_{38}H_{31}O_2[M+H]^+$ 519.2324, found 519.2302.

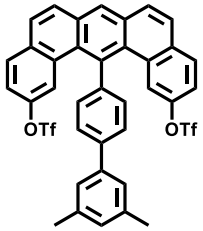
14-(3',5'-dimethyl-[1,1'-biphenyl]-4-yl)-dibenzo[a,j]anthracene-2,12-diol (12)



Compound **11** (250 mg, 0.48 mmol) was dissolved in 40 mL dry dichloromethane under argon atmosphere. Then, 5.8 mL 1 M BBr_3 (1.45 g, 5.78 mmol in dichloromethane) was added drop wise to the solution at 0 °C. The solution was then allowed to warm to room temperature and stirred for 6 h. The reaction was monitored by thin-layer chromatography plate. Once the reaction was completed, 10 mL water was slowly added at 0 °C. The mixture was washed with water and extracted with dichloromethane for three times. Then, the organic layer was dried over magnesium sulfate, and evaporated. The residue was re-precipitated from dichloromethane/hexane (1:50) and used directly for the next step without further purification.

Pale green powder.

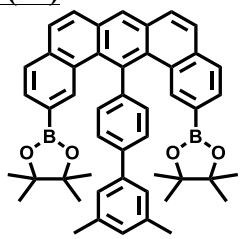
14-(3',5'-dimethyl-[1,1'-biphenyl]-4-yl)-dibenzo[a,j]anthracene-2,12-diylbis(trifluoromethanesulfonate) (13)



Crude compound **12** (236 mg, 0.48 mmol) was dissolved in 20 mL dichloromethane and cooled to 0 °C. Then 0.36 mL triethylamine (2.6 mmol) was added drop wise. Afterwards, 1 M Tf_2O (1.44 mL) solution was added by syringe. The mixture solution was allowed to warm to room temperature and stirred for 4 h. Once thin-layer chromatography plate showed the complete of the reaction, the dichloromethane was removed and the residue was purified by silica gel column chromatography (eluent: 10% ethyl acetate/hexane) yielded compound **13**.

White solid (Yield = 78%). Mp: 305.9-306.7 °C; ^{1}H NMR (300 MHz, CD_2Cl_2) δ 2.45 (s, 6H), 7.11 (s, 1H), 7.30 (d, J = 2.5 Hz, 2H), 7.38 (dd, J = 8.7, 2.5 Hz, 2H), 7.43 (s, 2H), 7.53 (d, J = 8.2 Hz, 2H), 7.79 (s, 1H), 7.82 (s, 1H), 7.92 (d, J = 2.8 Hz, 2H), 7.94 – 7.98 (m, 3H), 8.00 (s, 1H), 8.51 (s, 1H); ^{13}C NMR (75 MHz, CD_2Cl_2) δ 21.49, 116.39, 119.74, 122.06, 125.89, 127.94, 128.04, 128.83, 129.15, 129.64, 130.65, 130.87, 131.01, 132.46, 132.78, 134.23, 138.64, 139.91, 140.83, 142.22, 143.72, 146.60; HRMS (ESI, positive) m/z calcd for $C_{38}H_{25}F_6O_6S_2[M+H]^+$ 755.0997, found 755.0993.

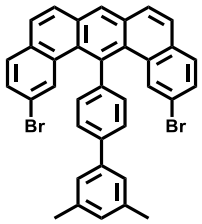
14-(3',5'-dimethyl-[1,1'-biphenyl]-4-yl)-dibenzo[a,j]anthracene-2,12-diyl-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (14)



A Schlenk tube was charged with compound **13** (110 mg, 0.145 mmol), [1,1'-bis(diphenylphosphino)ferrocene]dichloropalladium(II) (6 mg, 0.007 mmol), 5mL dry dioxane and triethylamine (0.12 mL, 0.87 mmol). The solution was degassed by argon bubbling for 10 min and pinacolborane (0.08 mL, 0.58 mmol) was added. The mixture was then heated at refluxing temperature for 12 h. Then the solvent was removed under vacuum and the residue was purified by silica gel chromatography (eluent: 5 % ethyl acetate/hexane) to afford compound **14**.

Light yellow solid (Yield = 71%). Mp: 282.6-283.2 °C; ^{1}H NMR (300 MHz, CD_2Cl_2) δ 1.02 (24H, d, J = 7.3 Hz), 2.40 (6H, d, J = 10.1 Hz), 7.01 (1H, s), 7.48 – 7.54 (4H, m), 7.69 – 7.73 (4H, m), 7.78 – 7.81 (2H, m), 7.85 – 7.88 (2H, d, J = 8.8 Hz), 7.96 – 7.99 (4H, m), 8.35 (1H, s); ^{13}C NMR (75 MHz, CD_2Cl_2) δ 21.70, 24.79, 83.97, 125.41, 128.02, 128.05, 128.50, 128.54, 128.96, 129.20, 129.36, 129.84, 131.21, 131.83, 132.06, 136.60, 136.78, 138.48, 140.45, 140.83, 142.87, 143.00, 143.22, 144.08; HRMS (APPI-TOF, positive) m/z calcd for $C_{48}H_{48}B_2O_4[M]^+$ 710.3739, found 710.3778.

2,12-dibromo-14-(3',5'-dimethyl-[1,1'-biphenyl]-4-yl)-dibenzo[a,j]anthracene (1a)

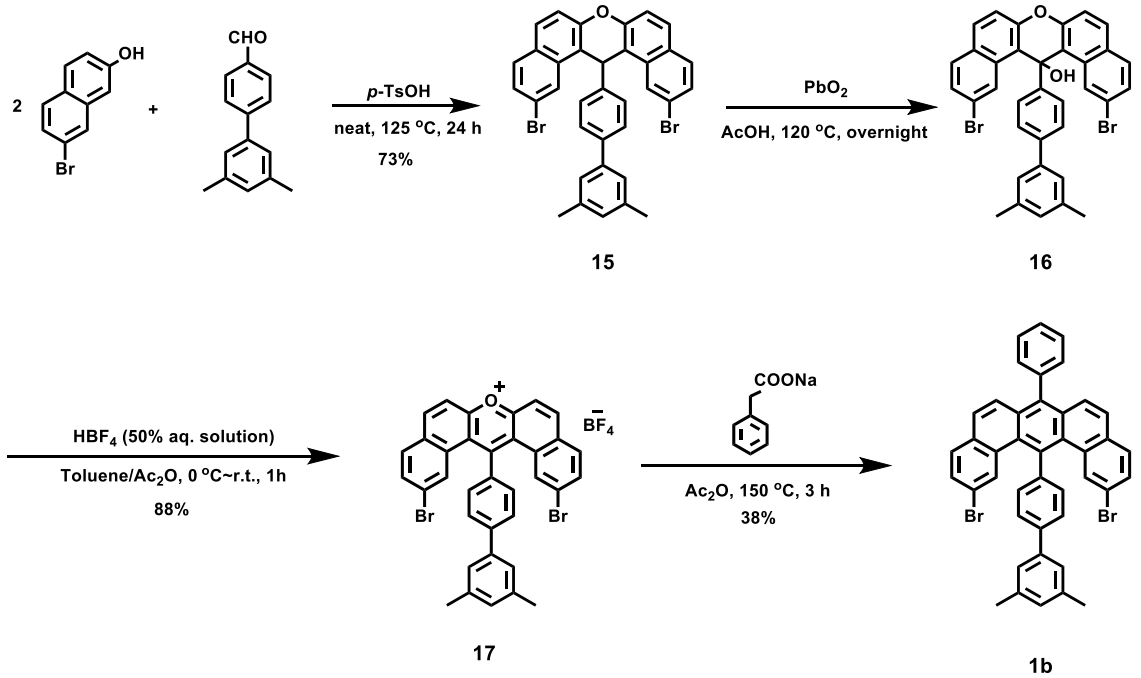


A 12 mL sealtube was charged with compound **14** (50 mg, 0.07 mmol), copper (II) bromide (95 mg, 0.42 mmol), 2 mL tetrahydrofuran, 6 mL methanol and 4 mL water. The tube was degassed by argon bubbling for 10 min, then sealed and heated at 120 °C overnight. After cooled to the room temperature, the mixture was extracted with dichloromethane for three times (5 mL × 3). The combined organic layer was dried over magnesium sulfate, and then evaporated. The residue was purified by silica gel chromatography (eluent: 10 % dichloromethane/hexane) and re-precipitated from dichloromethane/methanol (1:10) to yield final product **1a**.

Colorless solid (Yield = 60%). Mp: 267.9-268.5 °C; ^{1}H NMR (300 MHz, CD_2Cl_2) δ 2.45 (s, 6H), 7.10 (s, 1H), 7.47 – 7.55 (m, 8H), 7.69 – 7.73 (m, 4H), 7.88 – 7.97 (m, 4H), 8.43 (s, 1H); ^{13}C NMR (75 MHz, CD_2Cl_2) δ 21.79, 119.77, 125.96, 127.76, 128.03, 128.39, 128.70, 129.62, 129.80, 130.33, 130.48, 131.26, 132.02, 132.56, 132.71, 133.07, 133.37, 136.04, 139.05, 141.59, 143.46; HRMS (APPI-TOF, positive) m/z calcd for $C_{36}H_{24}Br_2[M]^+$ 614.0245, found 614.0246.

C. Synthetic route to monomer 1b

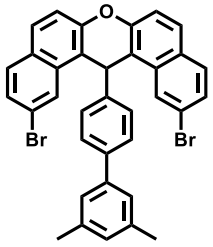
"U-shaped" monomer **1b** was synthesized based on the pyrylium chemistry, which was presented in Supplementary Scheme 2. First, compound **15** was prepared by condensation of 2-bromo-7-hydroxynaphthalene and corresponding aryl aldehyde (R = 3, 5-dimethylphenyl) under neat condition [12], followed by oxidation with lead (IV) oxide to afford compound **16**. Then, crude product of compound **16** was directly treated with tetrafluoroboric acid solution (48 wt. % in water) to afford pyrylium salt **17** [13,14]. Finally, after a condensation reaction of **17** with sodium 2-phenylacetate [15,16], target monomer **1b** was obtained in 38% yield.



Supplementary Scheme 2. General synthetic route towards monomer **1b**.

Experimental details and description

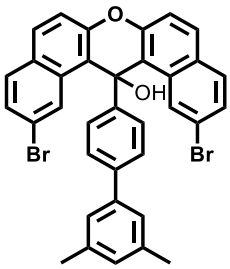
2,12-Dibromo-14-(3',5'-dimethyl-[1,1'-biphenyl]-4-yl)-14H-dibenzo[*a*,*j*]xanthene (15)
:



To a mixture of 3',5'-dimethyl-[1,1'-biphenyl]-4-carbaldehyde (2.36 g, 11.2 mmol) and 7-bromo-naphthol (5g, 22.4 mmol), *p*-toluenesulfonic acid monohydrate (*p*-TSA) (43 mg, 0.22 mmol) was added. The reaction mixture was stirred magnetically at 125 °C for 24 h. The reaction was monitored by thin-layer chromatography (TLC). Once the reaction was completed, the mixture was washed with EtOH–H_2O (1:3). The crude product was purified by recrystallization from ethanol to afford target compound **15**.

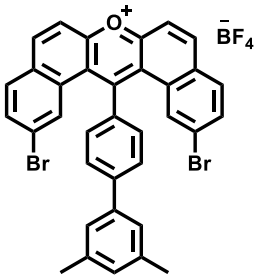
White needles (Yield = 73%); Mp: < 319 °C; ^{1}H NMR (500 MHz, $C_2D_2Cl_4$) δ 6.28 (s, 1H), 6.93 (s, 1H), 7.05 (s, 2H), 7.43 (d, J = 7.4 Hz, 2H), 7.53 (m, 6H), 7.77 (dd, J = 35.5, 8.7 Hz, 4H), 8.54 (s, 2H); ^{13}C NMR (125 MHz, $C_2D_2Cl_4$) δ 21.27, 37.51, 37.51, 115.91, 118.46, 121.47, 124.82, 125.01, 127.42, 127.72, 128.21, 128.88, 128.95, 129.30, 130.36, 132.33, 138.08, 139.53, 139.91, 142.84, 148.98; HRMS (MALDI-TOF, positive) *m/z* calcd for $C_{35}H_{24}Br_2O[M]^+$ 620.0173, found 620.0178.

2,12-Dibromo-14-(3',5'-dimethyl-[1,1'-biphenyl]-4-yl)-14H-dibenzo[*a*,*j*]xanthen-14-ol (16)



Compound **15** (6.6 g, 10.6 mmol) and lead dioxide (PbO_2) (3.8 g, 15.9 mmol) in glacial acetic acid (50 mL) were stirred while heating on an oil bath at 120 °C for 12 h. The cooled mixture was poured into crushed ice and the solid residue **16** (crude compound) was filtered off, dried under vacuum at 80 °C and used directly for the next step.

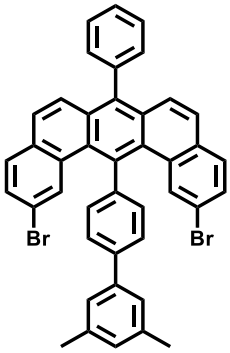
2,12-Dibromo-14-(3',5'-dimethyl-[1,1'-biphenyl]-4-yl)-14-dibenzo[*a,j*]xanthenylium tetrafluoroborate (17)



Compound **16** (2 g, 3.1 mmol) in acetic anhydride (15 ml) and toluene (10 mL) was cooled to 0 °C and treated with tetrafluoroboric acid solution (48 wt. % in water) (3.91 mL, ca. 31 mmol) until no further precipitation occurred. The cooled solution was filtered and washed with anhydrous ether to yield **17**.

Orange red powder (yield=88%). ^{1}H NMR (500 MHz, $C_2D_2Cl_4$) δ 2.50 (s, 6H), 7.20 (s, 1H), 7.46 (s, 2H), 7.52 (s, 2H), 7.61 (d, J = 7.8 Hz, 2H), 7.95 (d, J = 8.3 Hz, 2H), 8.08 (d, J = 8.4 Hz, 2H), 8.20 (d, J = 7.8 Hz, 2H), 8.32 (d, J = 9.0 Hz, 2H), 8.81 (d, J = 9.1 Hz, 2H); ^{13}C NMR (125 MHz, $C_2D_2Cl_4$) δ 21.47, 117.54, 121.13, 125.45, 126.18, 126.58, 129.74, 130.42, 130.93, 131.30, 131.45, 132.59, 133.07, 135.37, 138.92, 139.37, 146.37, 146.90, 159.45, 167.67; HRMS (MALDI-TOF, positive) *m/z* calcd for $C_{35}H_{23}Br_2O^+[M]^+$ 617.0116, found 617.0127.

2,12-Dibromo-14-(3',5'-dimethyl-[1,1'-biphenyl]-4-yl)-7-phenyl-dibenzo[*a,j*]anthracene (1b)



A mixture of the pyrylium salt **17** (4.2g, 5.9 mmol) and sodium 2-phenylacetate (2.8 g, 17.8 mmol) in acetic anhydride (Ac_2O, 50 mL) was stirred at 150 °C for 12 h under argon

atmosphere. After cooled to room temperature, the precipitate was filtered off and washed with acetic anhydride, then methanol. The crude product was re-precipitated from chloroform and hexane to give target monomer **1b**.

Yellow powder (yield= 38%). Mp: > 400 °C; ^{1}H NMR (500 MHz, $C_2D_2Cl_4$) δ 2.48 (s, 6H), 7.12 (s, 1H), 7.48 (d, J = 7.3 Hz, 6H), 7.56 – 7.50 (m, 6H), 7.57 (s, 2H), 7.70 – 7.60 (m, 5H), 7.91 (d, J = 8.0 Hz, 2H); ^{13}C NMR (125 MHz, $C_2D_2Cl_4$) δ 21.48, 118.57, 120.18, 125.62, 125.80, 126.85, 126.97, 127.79, 128.53, 129.04, 129.11, 129.73, 130.30, 131.19, 131.71, 132.00, 132.10, 132.60, 137.39, 137.77, 138.33, 138.83, 141.17, 142.30, 142.67; HRMS (MALDI-TOF, positive) m/z calcd for $C_{42}H_{28}Br_2[M]^+$ 690.0558, found 690.0561.

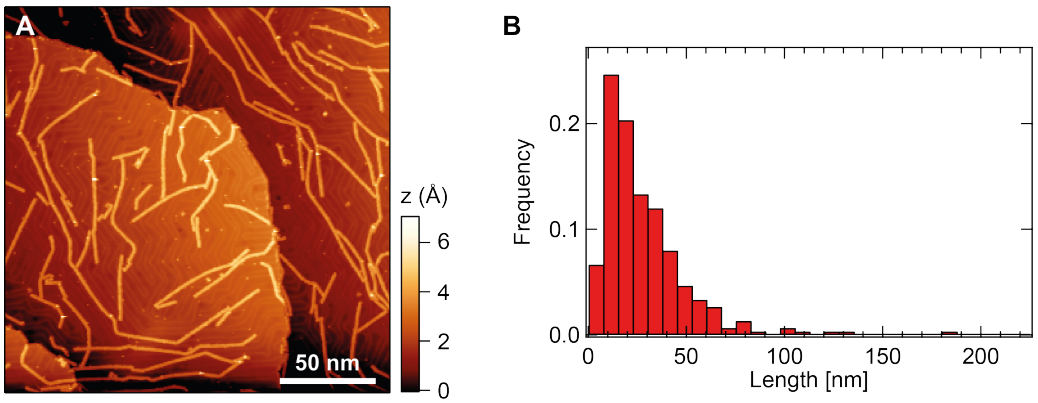


Fig. S1 Length analysis of 6-ZGNRs grown on Au(111). (**A**) Characteristic large scale constant current STM image used for the determination of the ribbon length distribution (T = 5 K, 200 nm × 200 nm, U = -1.0 V, I = 1 nA). (**B**) Ribbon length distribution based on the analysis of three large scale STM images with a total of 300 ribbons.

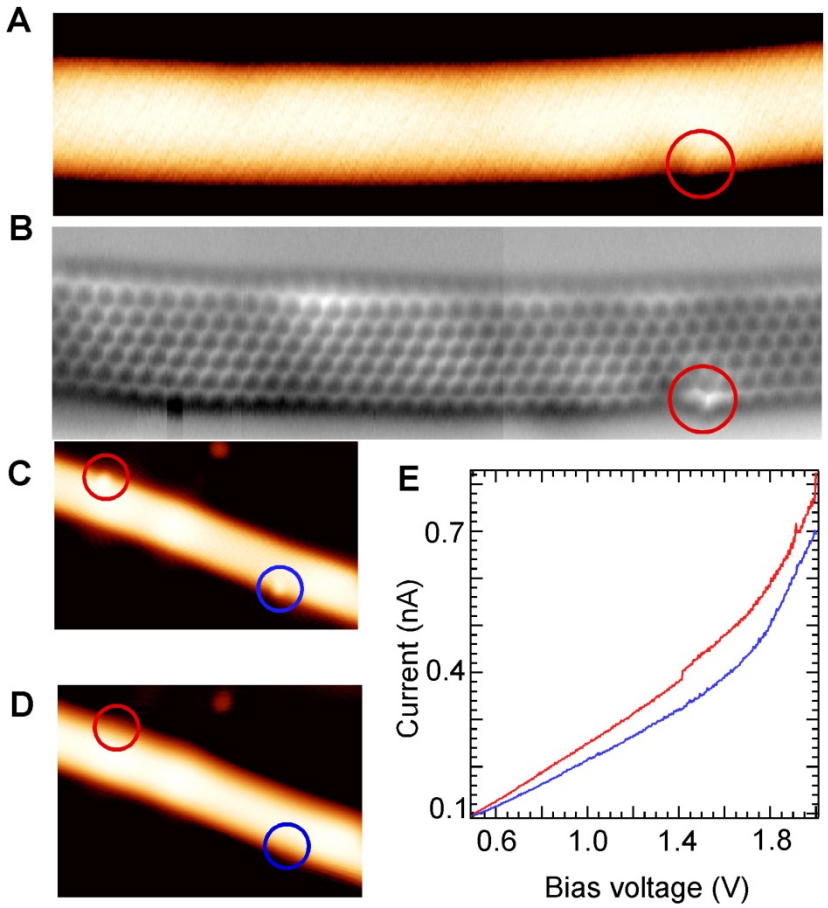


Fig. S2. H_2 defects of 6-ZGNRs. (**A**) Constant-current STM image (9.5 nm x 2.8 nm, T = 5 K, U = -1.0 V, I = 20 pA) of 6-ZGNR showing a H_2 defect marked by red circles. (**B**) Constant-height AFM frequency-shift image (9.5 nm x 3 nm, oscillation amplitude A = 0.7 Å, sample voltage V = 10 mV) taken at the same place as in (A). (**C**) STM image (10 nm x 5 nm, T = 5 K, U = -1.0 V, I = 20 pA) of 6-ZGNR showing two H_2 defects. (**D**) STM image (10 nm x 5 nm, T = 5 K, U = -1.0 V, I = 20 pA) showing the same ribbon in (C) after tip-induced dehydrogenation. (**E**) Current-voltage spectra taken above H_2 defects to remove one hydrogen.

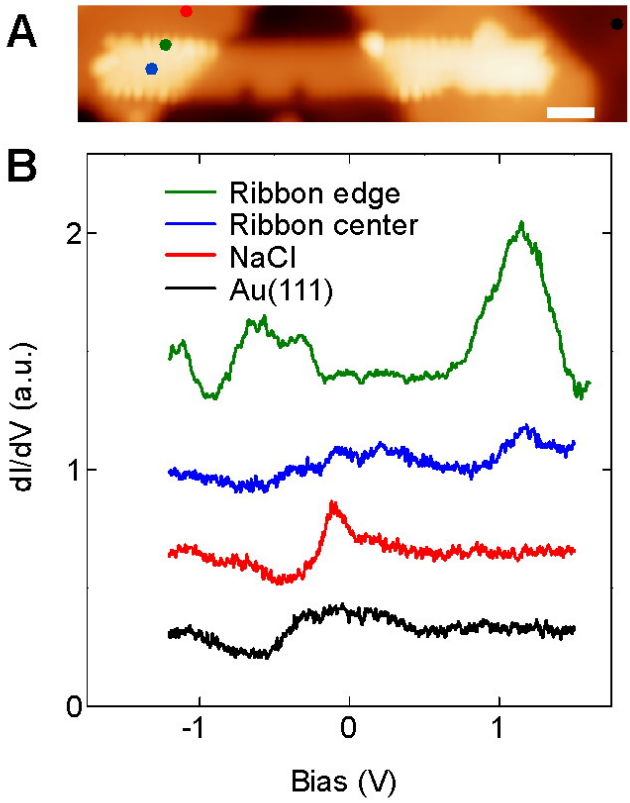


Fig. S3. dI/dV spectra recorded at different positions. (**A**) Constant-current STM image (T = 5 K, U = -0.25 V, I = 100 pA) of a partially decoupled 6-ZGNR. Scale bar: 1nm. (**B**) Differential conductance (dI/dV) spectra recorded at nearby Au(111) (black), NaCl monolayer (red), ribbon center (green), and zigzag edge (green) as indicated by colored dots in (**A**)), all acquired using the same tip.

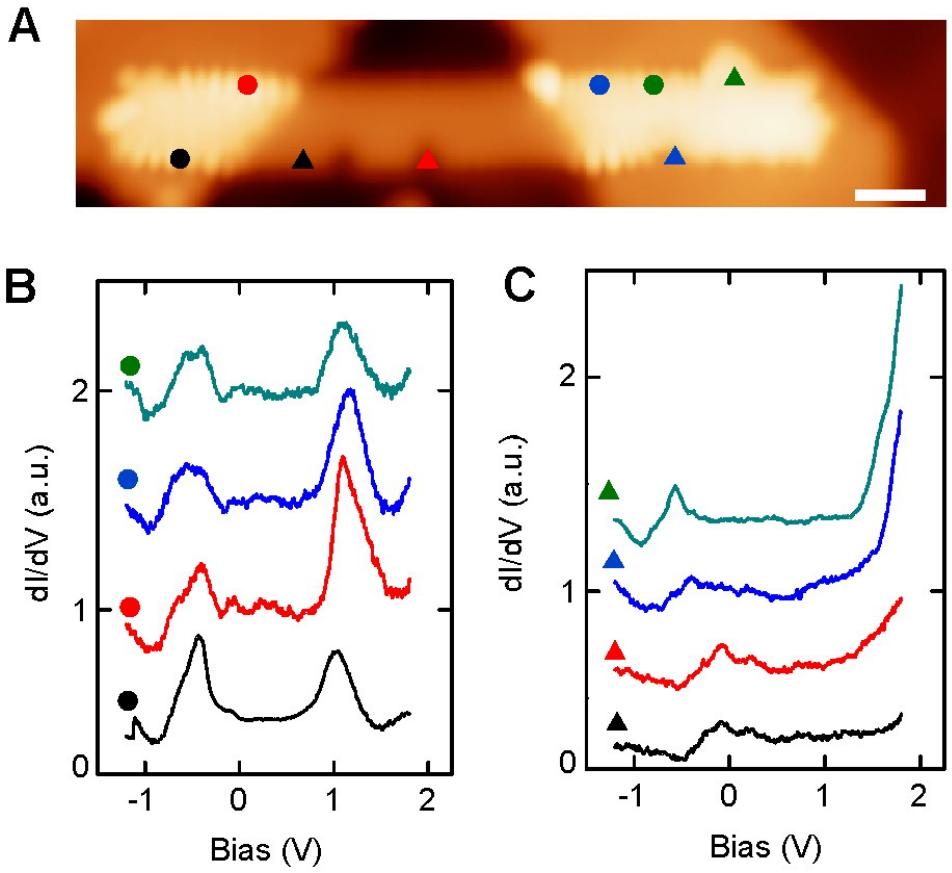


Fig. S4. dI/dV spectra recorded at different positions within a partially decoupled ZGNR. (**A**) Constant-current STM image (T = 5 K, U = -0.25 V, I = 100 pA) of a partially decoupled 6-ZGNR. Scale bar: 1nm. (**B**) Differential conductance (dI/dV) spectra recorded at decoupled zigzag edges (positions marked by colored dots in (**A**)). (**C**) Differential conductance (dI/dV) spectra recorded at zigzag edges on Au(111), a missing phenyl ring defect on Au(111), a missing phenyl ring defect on NaCl, and a protrusion defect of unknown nature (positions indicated by colored triangles in (**A**)). All spectra recorded at electronically decoupled zigzag edges (6-ZGNR on NaCl) show identical spectroscopic features deriving from the edge states, while the features are totally/partially missing for the other cases.

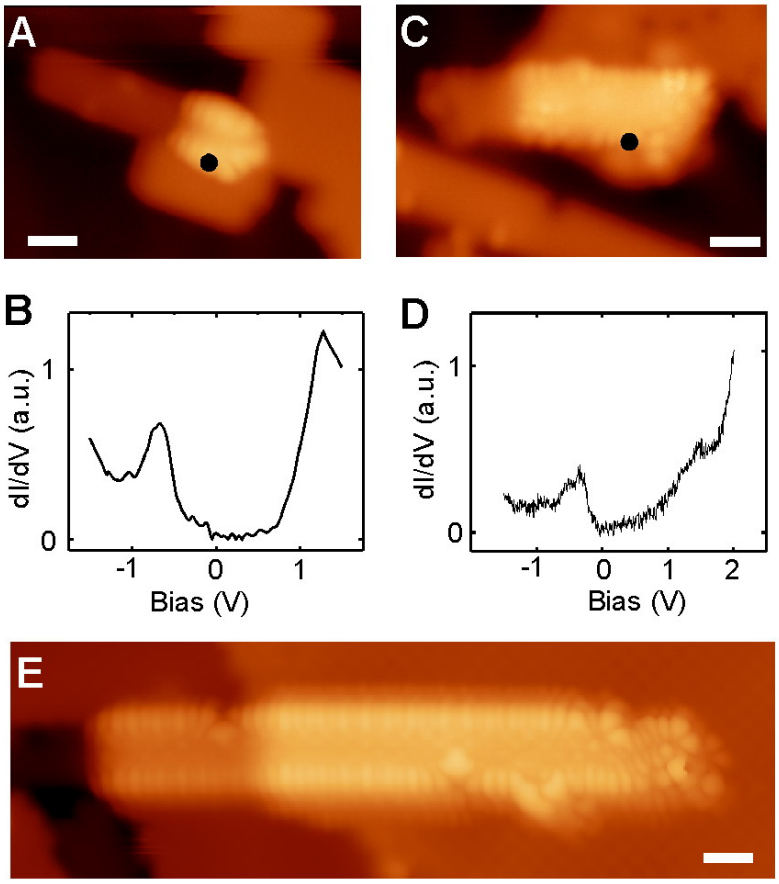


Fig. S5. Other examples of partially decoupled ZGNRs. (**A, C**) Constant-current STM image (T = 5 K, U = -1 V, I = 50 pA) of two partially decoupled 6-ZGNRs. (**B, D**) Differential conductance (dI/dV) spectra recorded at a decoupled zigzag edge as marked by black dots in (**A**) and (**C**), respectively. (**E**) Constant-current STM image (T = 5 K, U = -0.7 V, I = 5 pA) showing an almost fully decoupled 6-ZGNR (the left end is in contact with the metal substrate, the remaining part of the ribbon is on monolayer NaCl and on bilayer NaCl (right part), respectively). In contrast to the other cases presented, this ribbon has not been moved onto the NaCl by STM tip manipulation, but has "accidentally" been (partially) undergrown by NaCl islands upon high coverage NaCl deposition. For the case of STM manipulation, we find that the success rate for releasing the ribbon from the STM tip after lateral manipulation is significantly higher if one part of the ribbon remains on the metal. With the applied manipulation strategy, we were unable to stabilize a ribbon fully on a NaCl island. All scale bars: 1 nm.

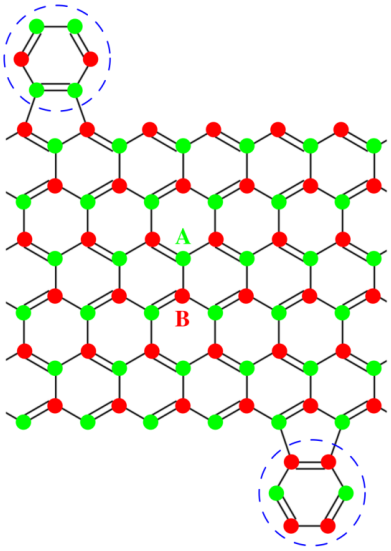


Fig. S6. Geometry frustration at the zigzag backbone. Sub-lattice description of graphene, illustrated for the case of the edge-modified 6-ZGNR. Each atom on the A sub-lattice is surrounded by three B sub-lattice atoms and vice-versa. The blue dotted-circles indicate frustrated arrangement of sublattices due to the fluoranthene subunits.

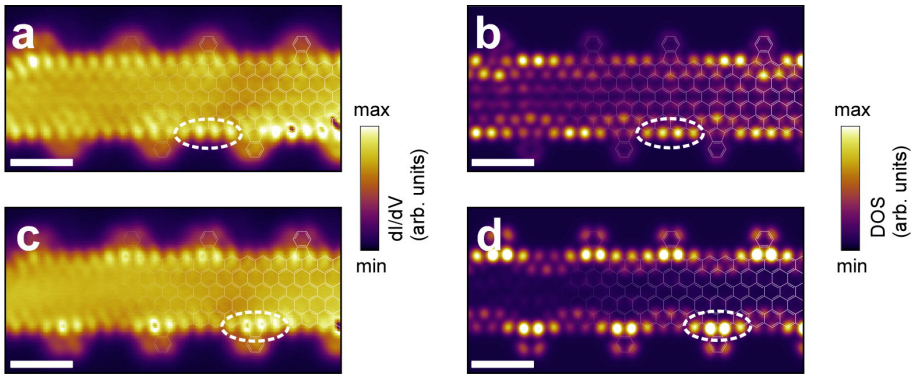
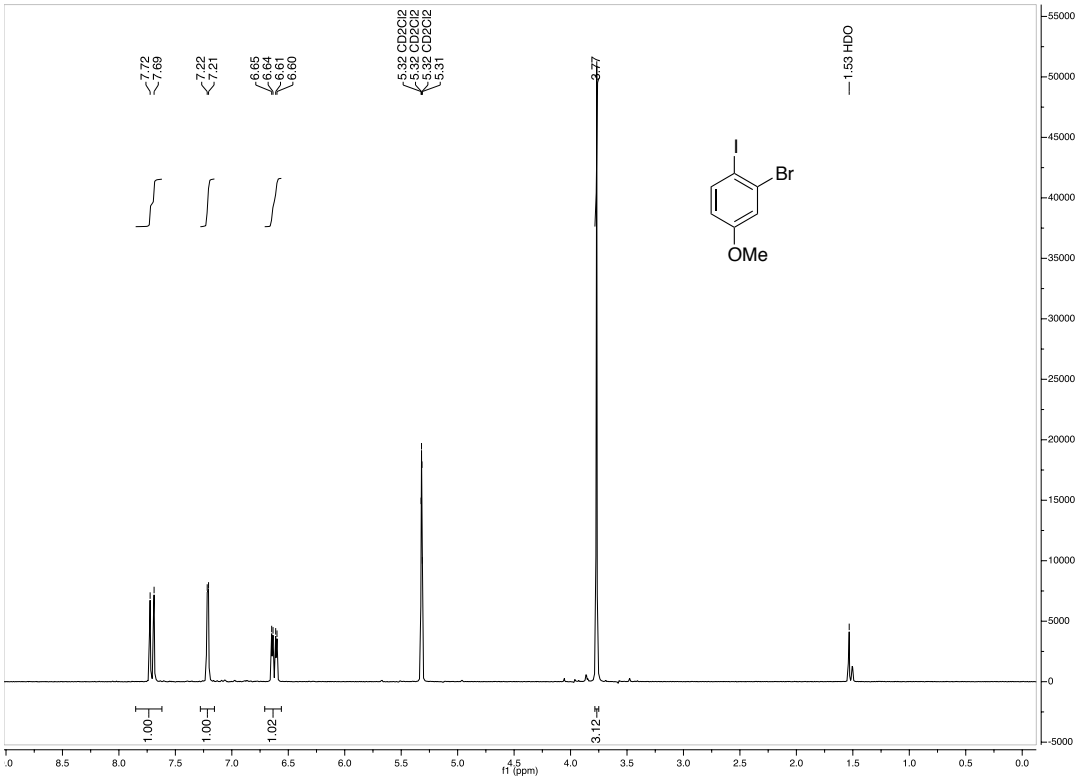
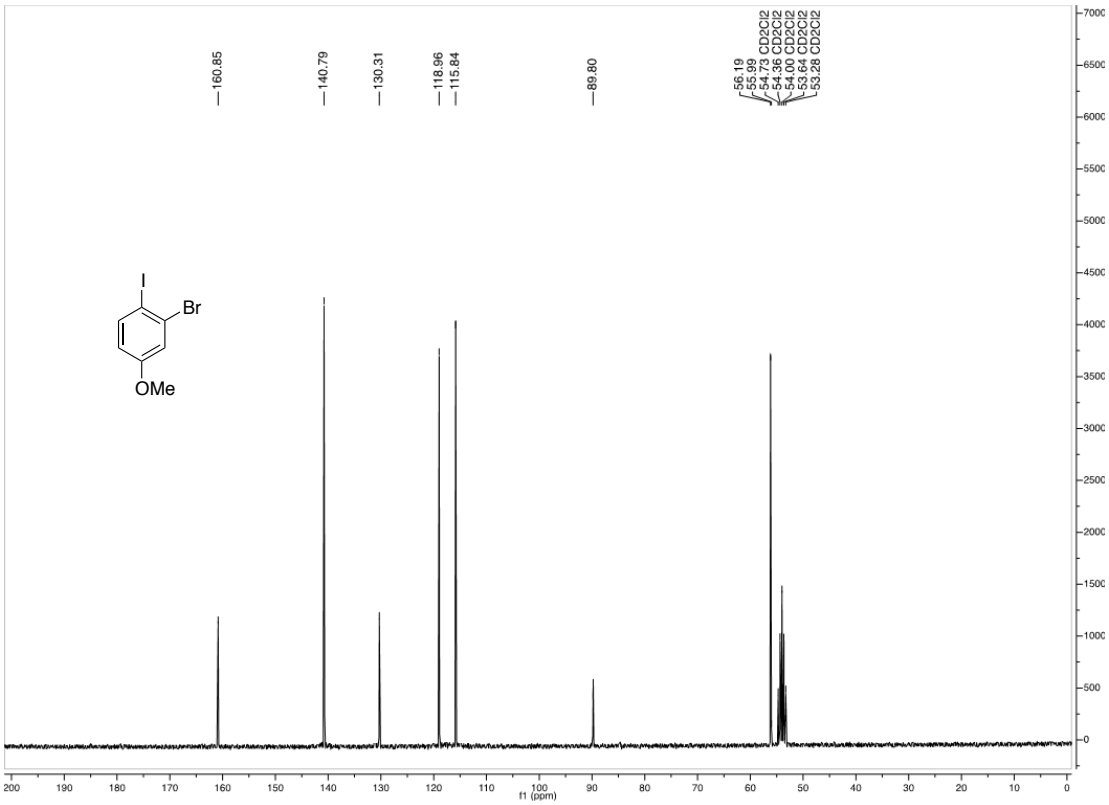


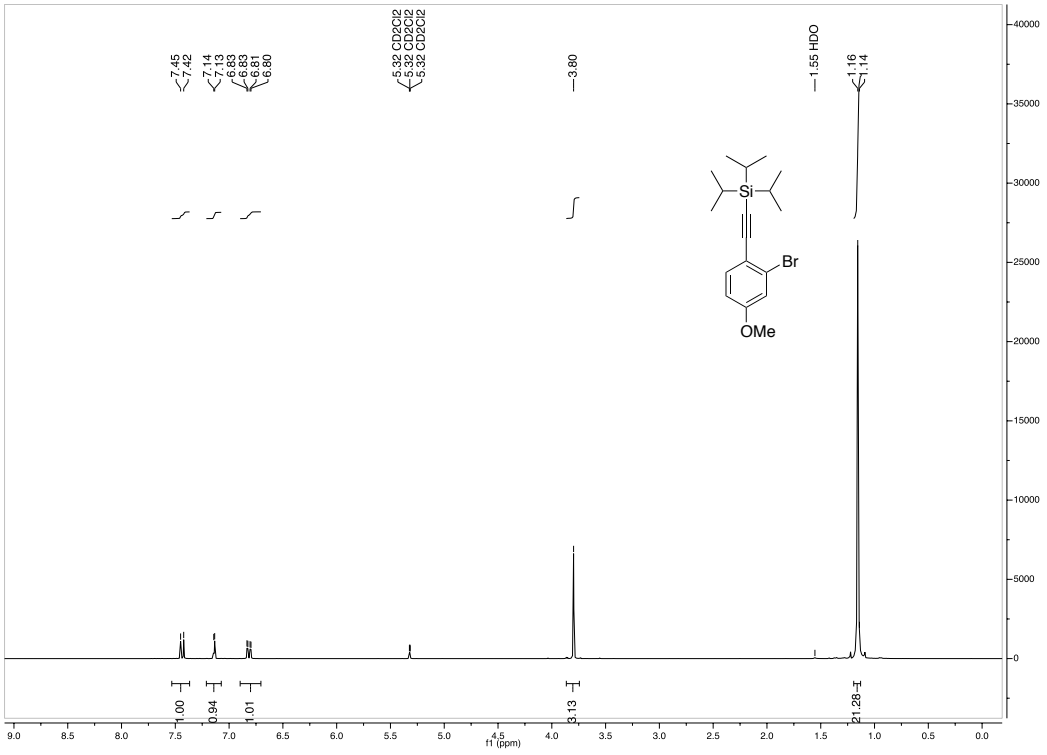
Fig. S7. dI/dV maps and DFT-based density of states distributions of edge-modified 6-ZGNRs. (**a**), Constant height dI/dV map of the occupied states (U = -0.15 V). (**b**), DFT-based density of states at E = -0.10 eV (corresponding to the highest occupied state). (**c**), Constant height dI/dV map of the unoccupied states (U = 0.15 V). **d**, DFT-based density of states at E = 0.15 eV (corresponding to the lowest unoccupied level. Dashed ovals in (**a** – **d**) highlight the zigzag segments with the most prominent contributions for occupied and unoccupied states, respectively. All scale bars: 1 nm.

In contrast to pristine 6-ZGNRs, the metal-adsorbed edged-modified 6-ZGNRs reveal increased intensity along the ZGNR edges. While the edge states at pristine zigzag edges occupy exclusively one carbon sublattice (*e.g.* sublattice A), in the case of the edge-modified 6-ZGNR the five-membered ring of the fluoranthene subunit locally disturbs the bipartite character of the graphene lattice by directly connecting carbon atoms belonging to the same sublattice (Supporting Figure S6). This topological defect breaks translational symmetry along the zigzag edge and gives rise to a linear combination of Bloch states with maximum amplitude in between the defects (a, b) and maximum amplitude on the defects (c, d).

^{1}H and ^{13}C NMR spectra



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7.13
6.83
6.83
6.81
6.80
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5.32 CD2Cl2
5.32 CD2Cl2
3.80
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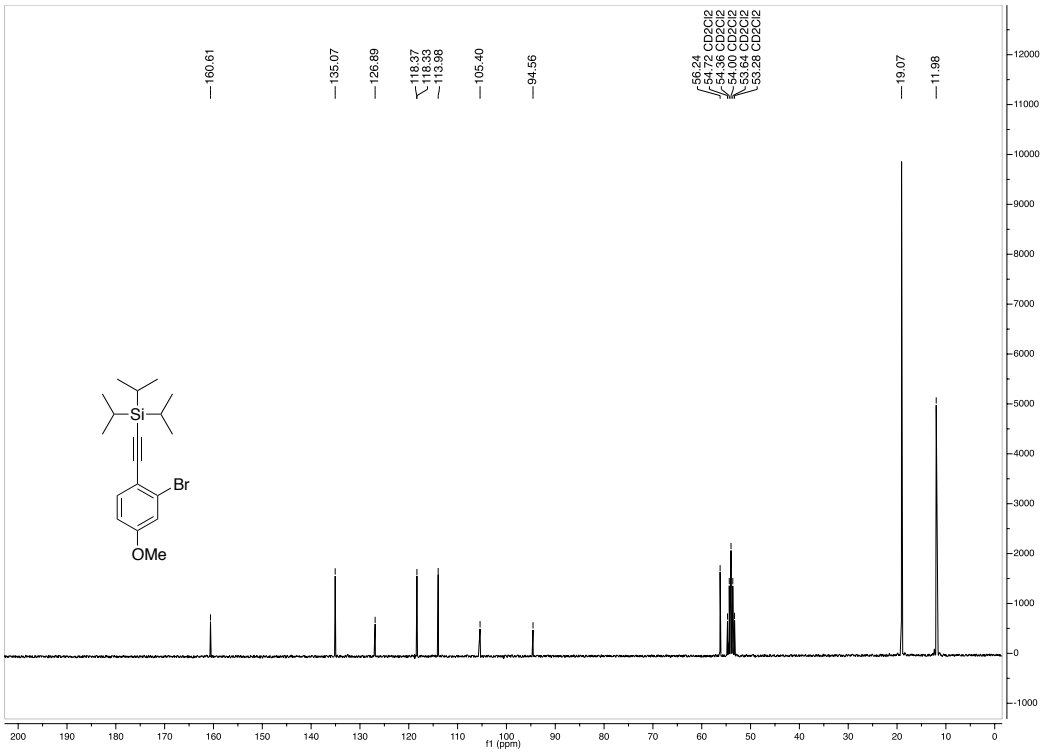
Br
OMe
1.00
0.94
1.01
3.13
21.28
f1 (ppm)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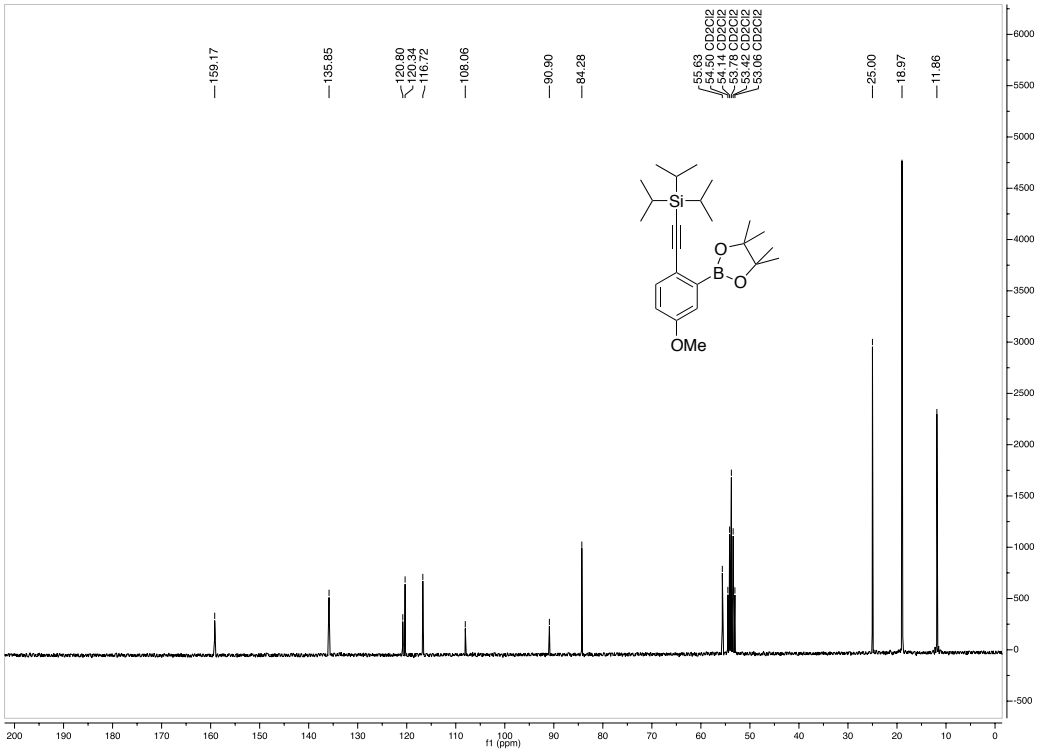
f1 (ppm)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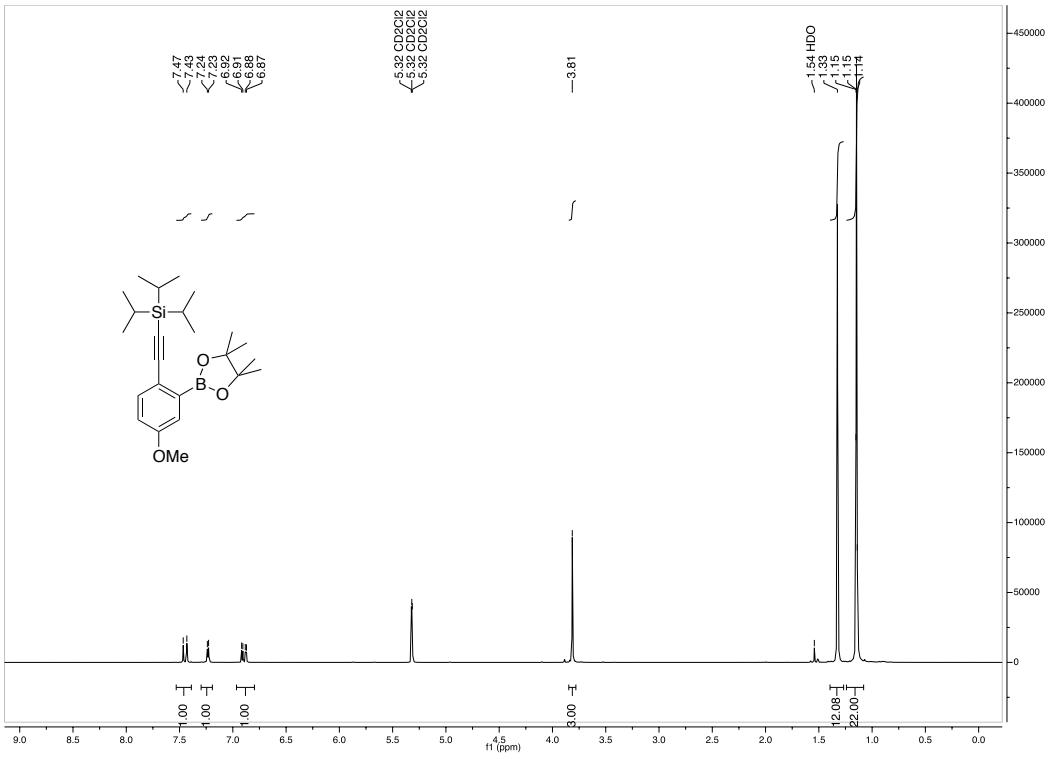
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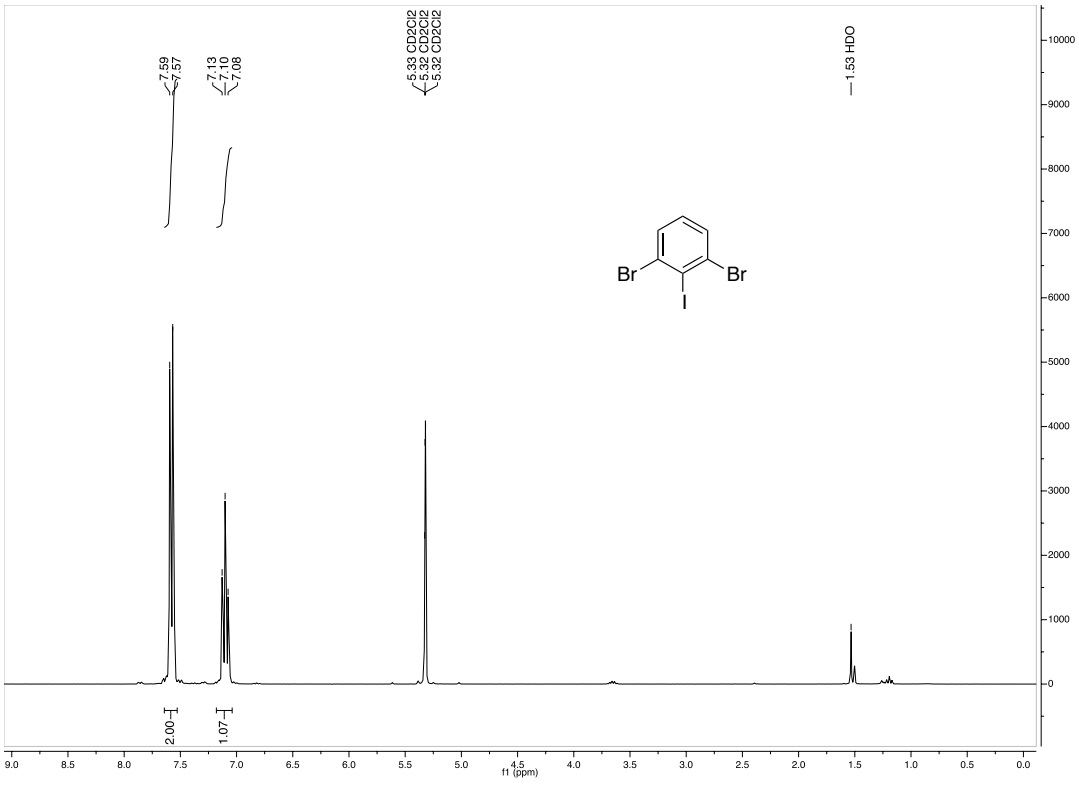
f1 (ppm)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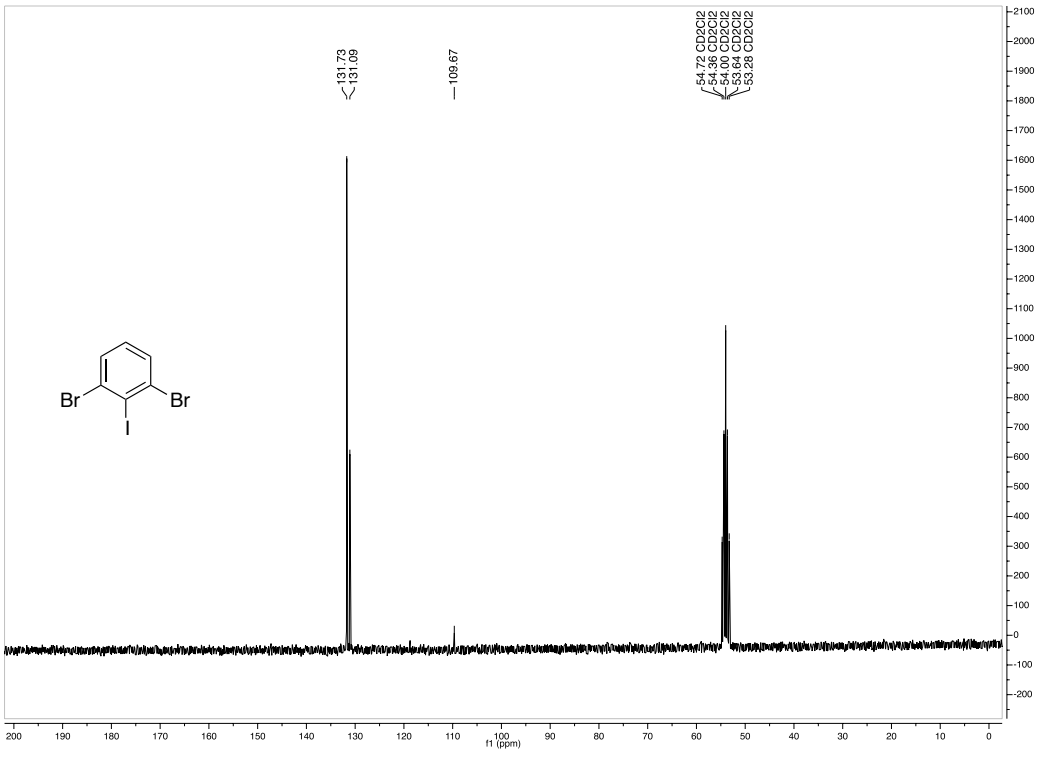
f1 (ppm)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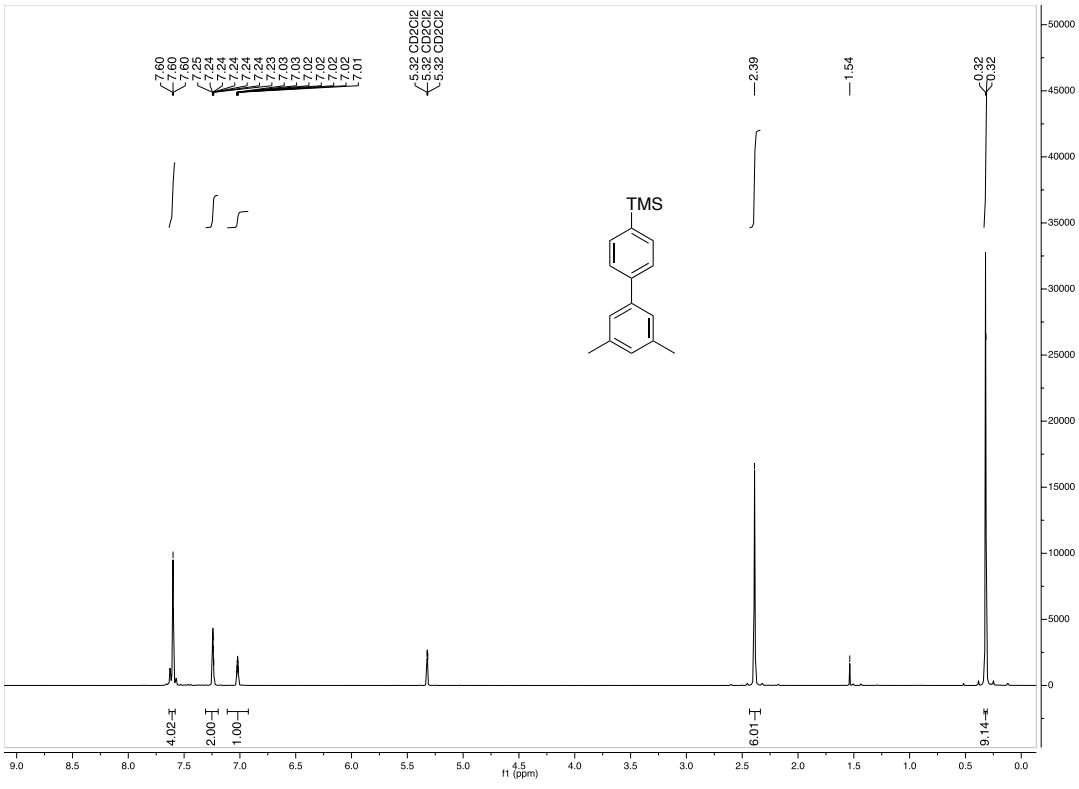
f1 (ppm)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TMS
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2.00
1.00
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9.14
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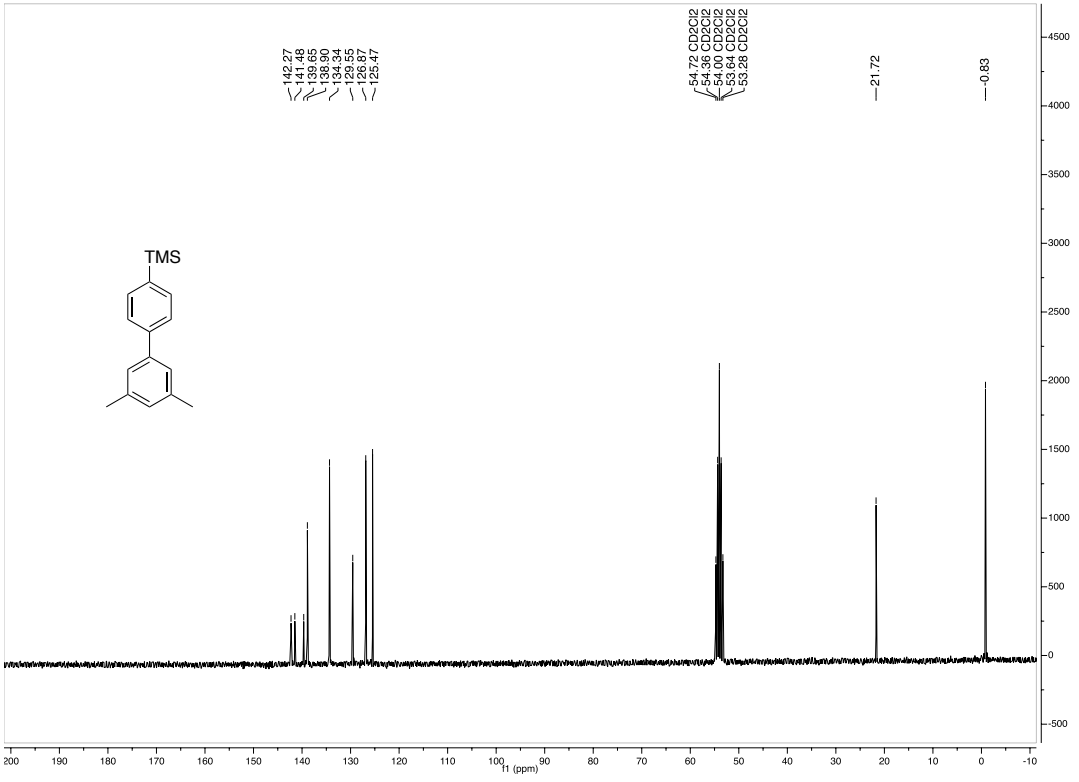
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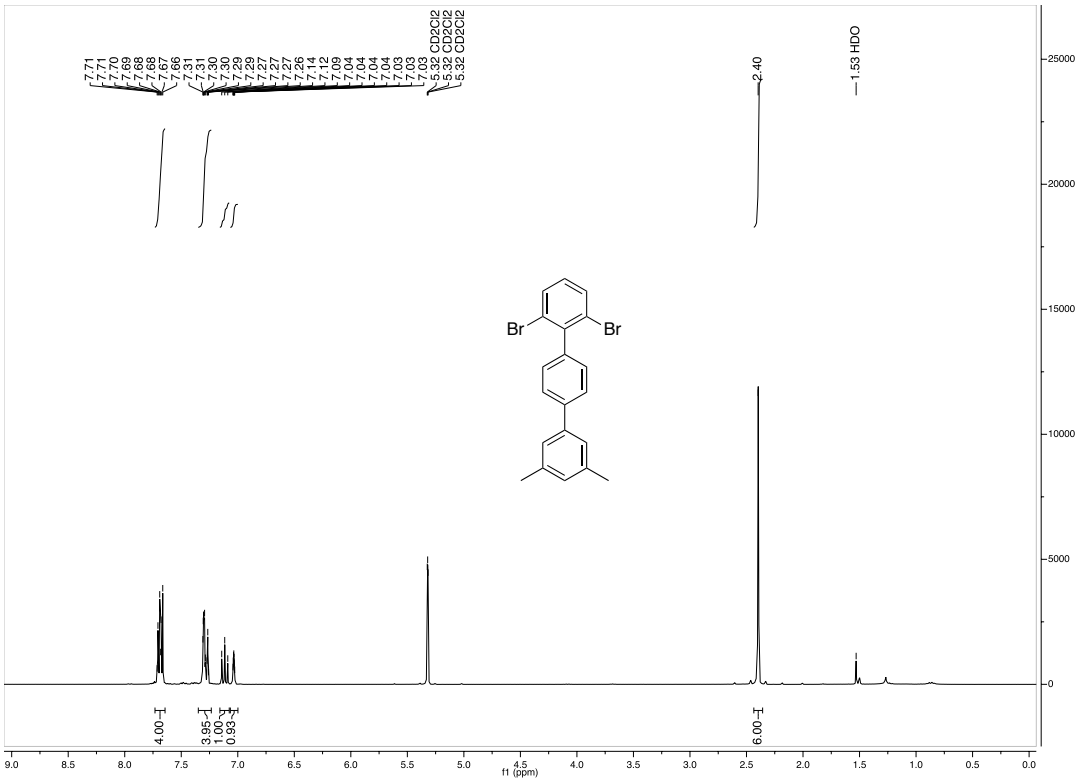
f1 (ppm)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7.27
7.27
7.27
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7.14
7.12
7.09
7.04
7.04
7.04
7.04
7.03
7.03
7.03
5.33 CD2Cl2
5.32 CD2Cl2
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Br
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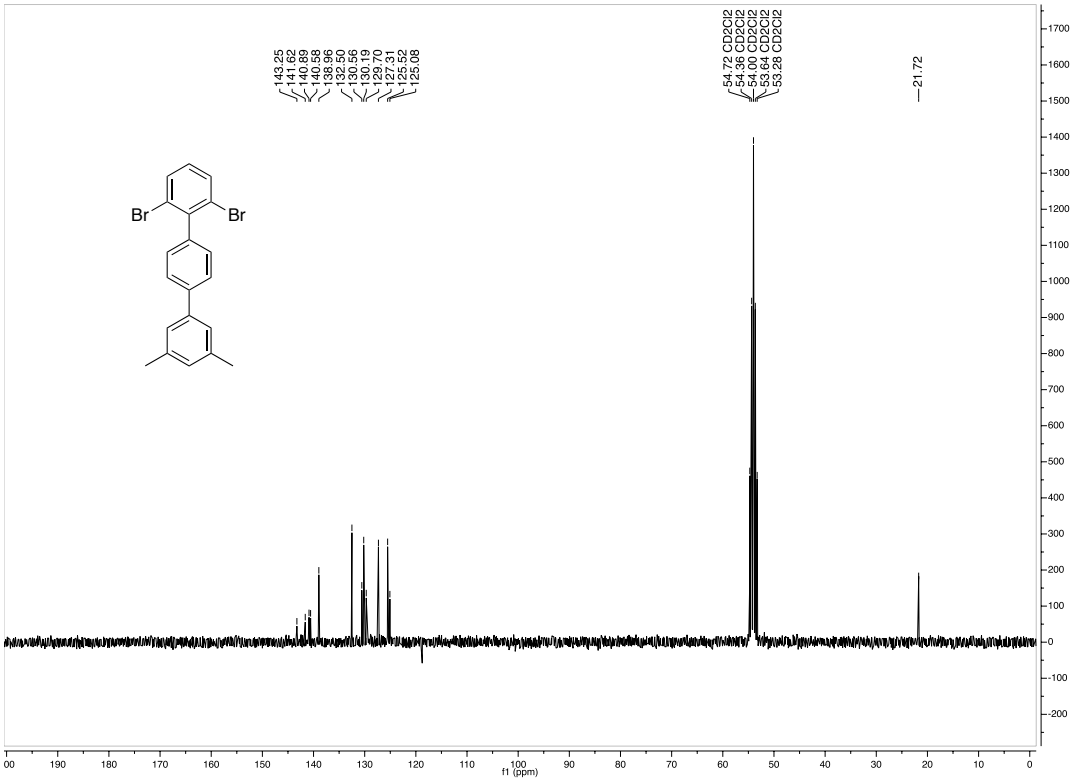
f1 (ppm)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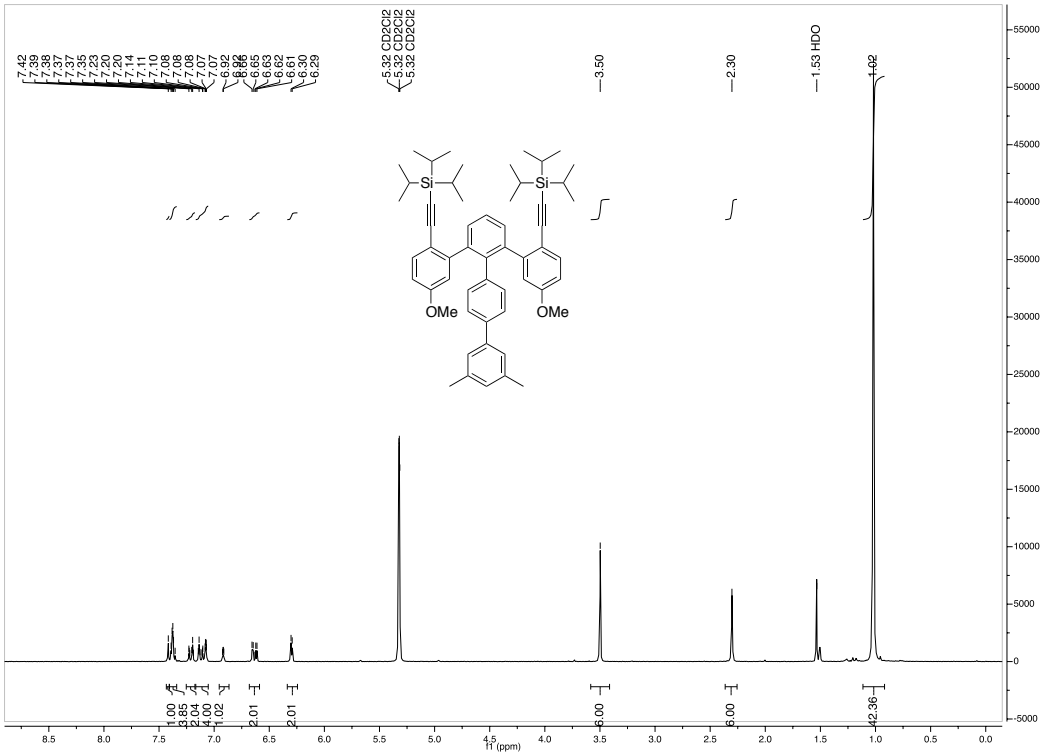
f1 (ppm)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7.08
7.08
7.07
7.07
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6.92
6.65
6.63
6.62
6.61
6.30
6.29
5.32 CD2Cl2
5.32 CD2Cl2
5.32 CD2Cl2
3.50
2.30
1.53 HDO
1.02
Si
Si
OMe
OMe
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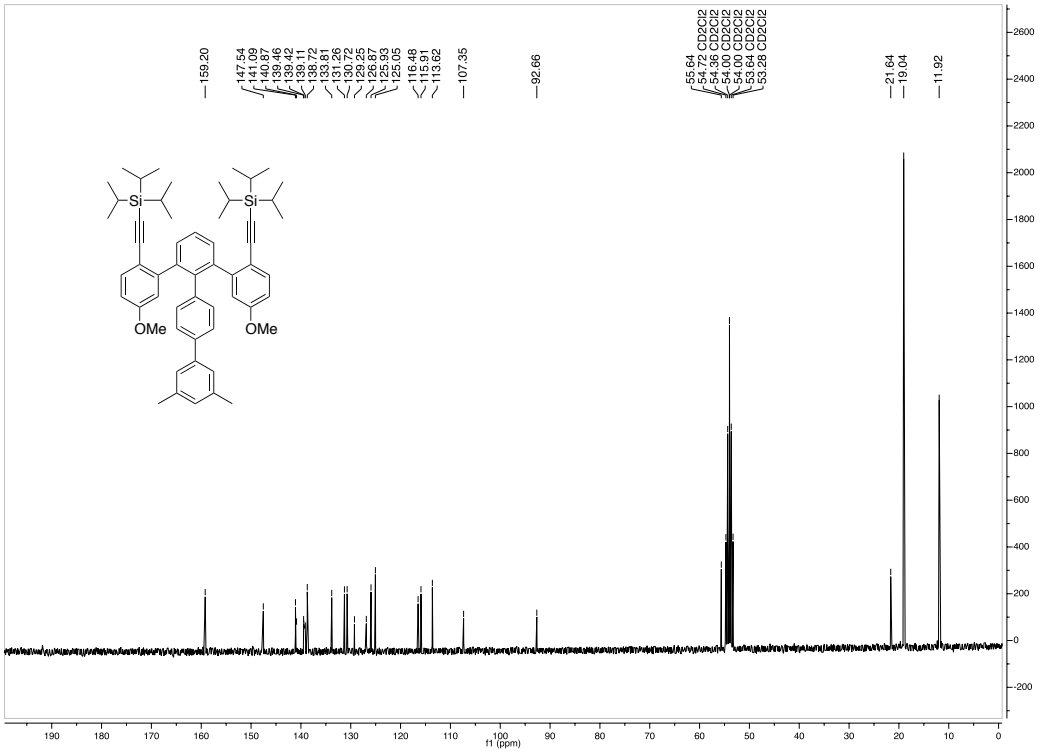
f1 (ppm)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107.35
92.66
55.64
54.72 CD2Cl2
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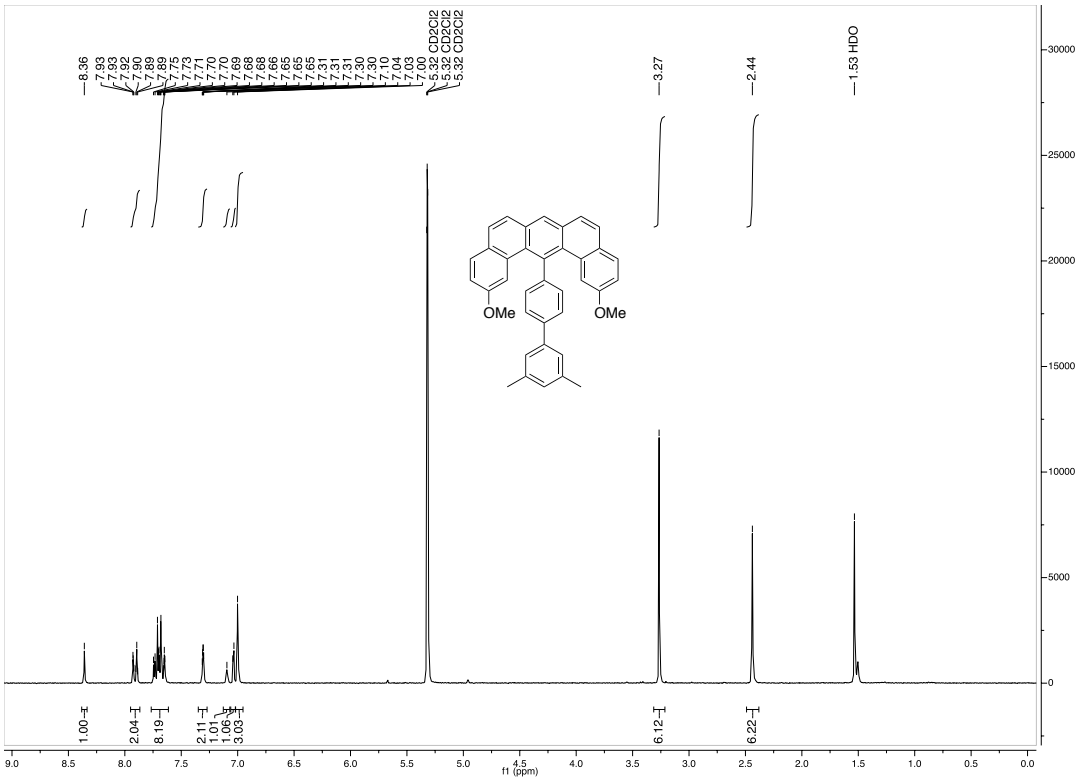
11.92
Si
Si
OMe
OMe
f1 (ppm)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7.30
7.10
7.04
7.03
5.32 CD2Cl2
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5.32 CD2Cl2
3.27
2.44
1.53 HDO
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3.00
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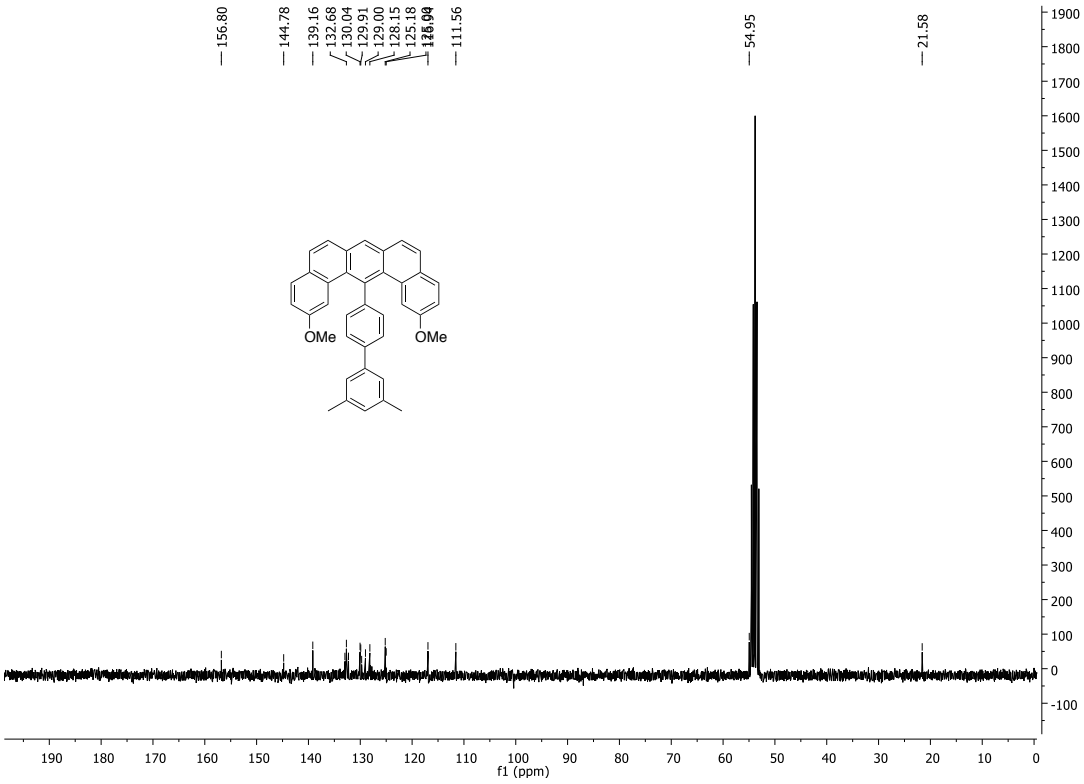
f1 (ppm)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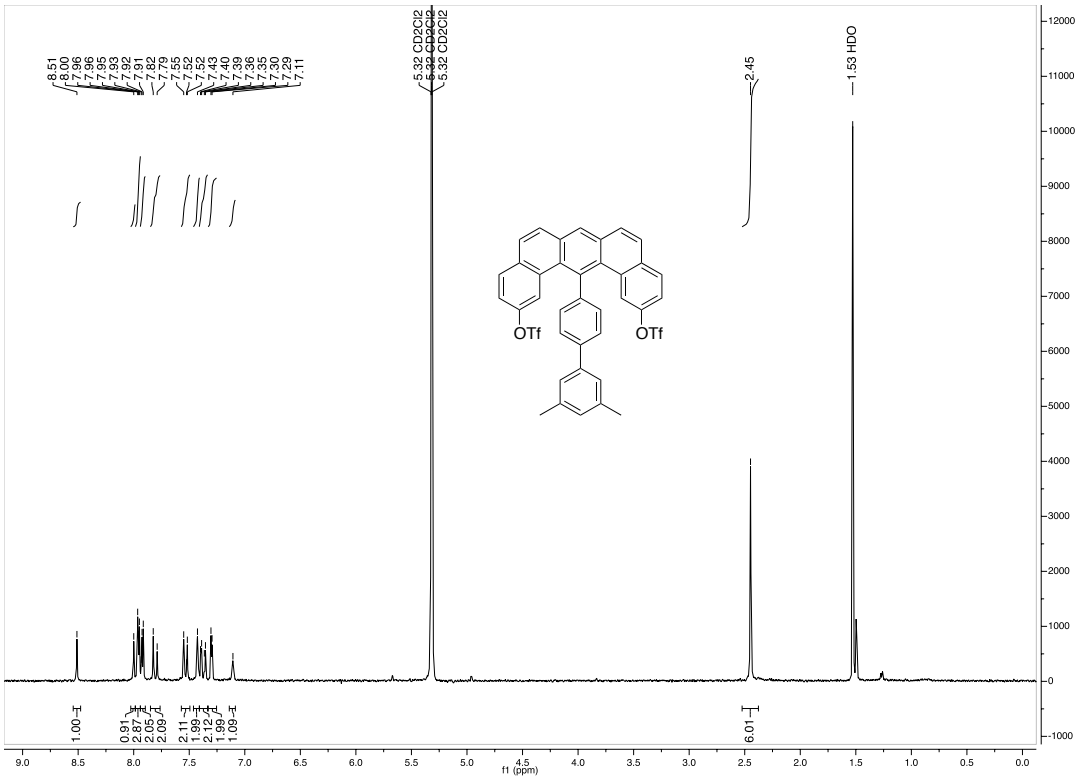
OMe
OMe
f1 (ppm)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7.29
7.11
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5.32 CD2Cl2
5.32 CD2Cl2
2.45
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OTf
OTf
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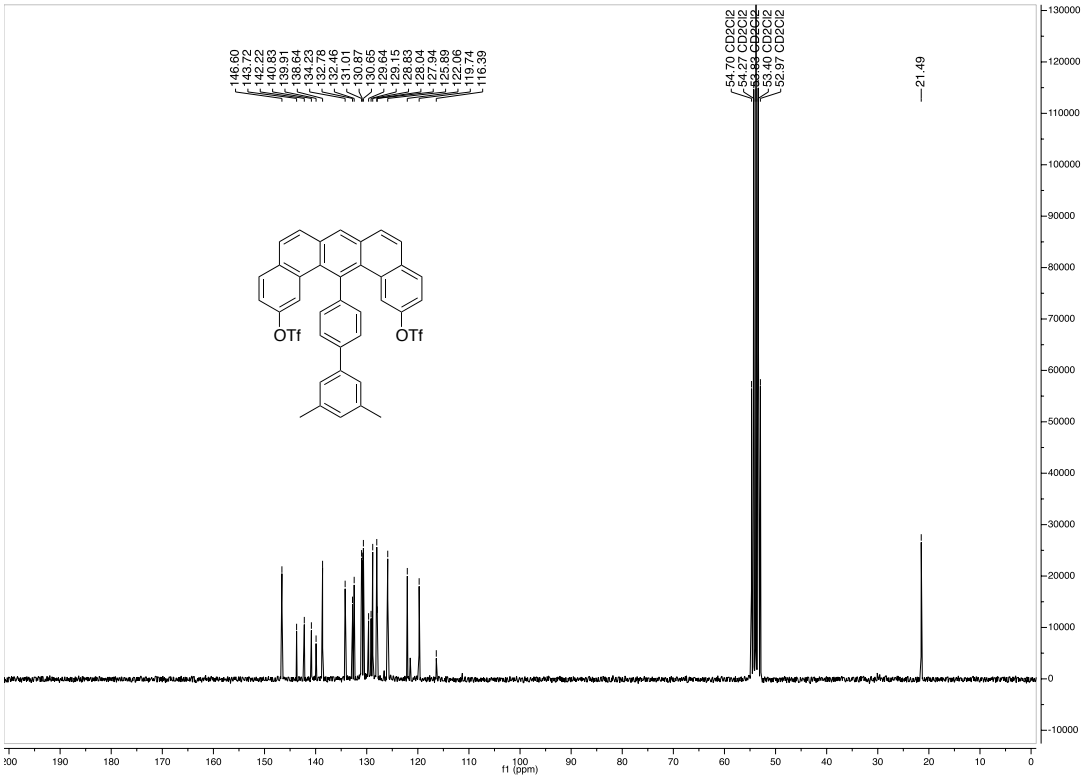
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129.15
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128.04
127.94
125.89
122.06
119.74
116.39
54.70 CD2Cl2
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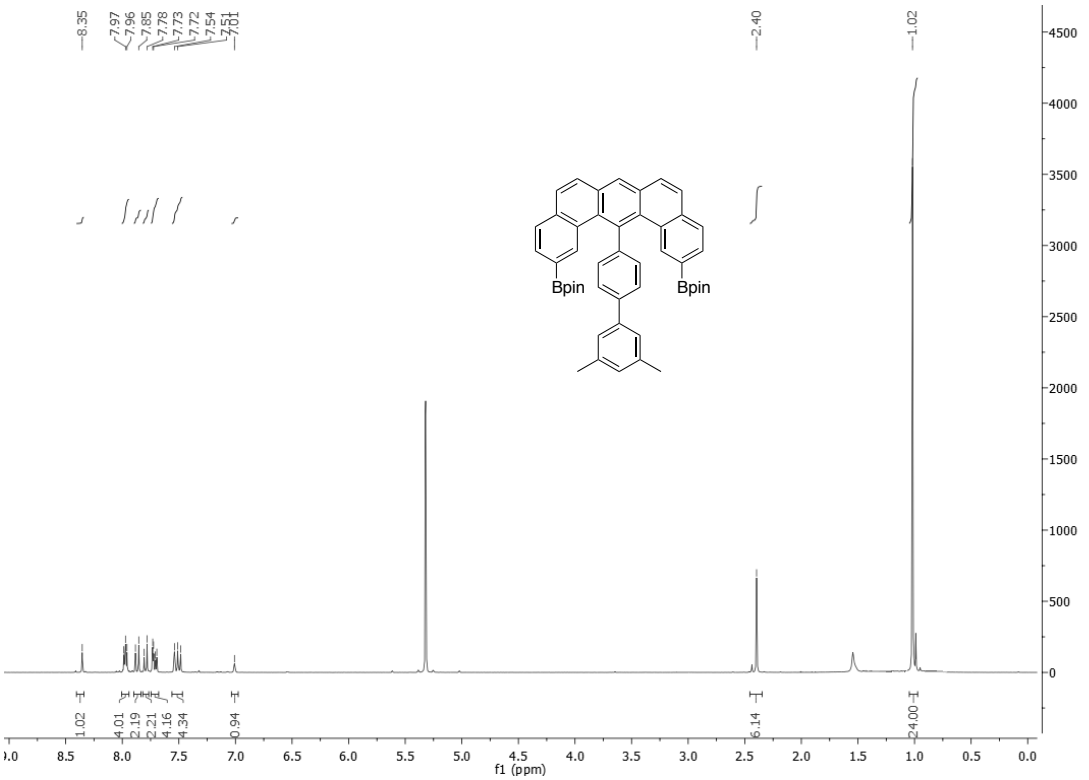
OTf
OTf
f1 (ppm)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7.51
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1.02
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Bpin
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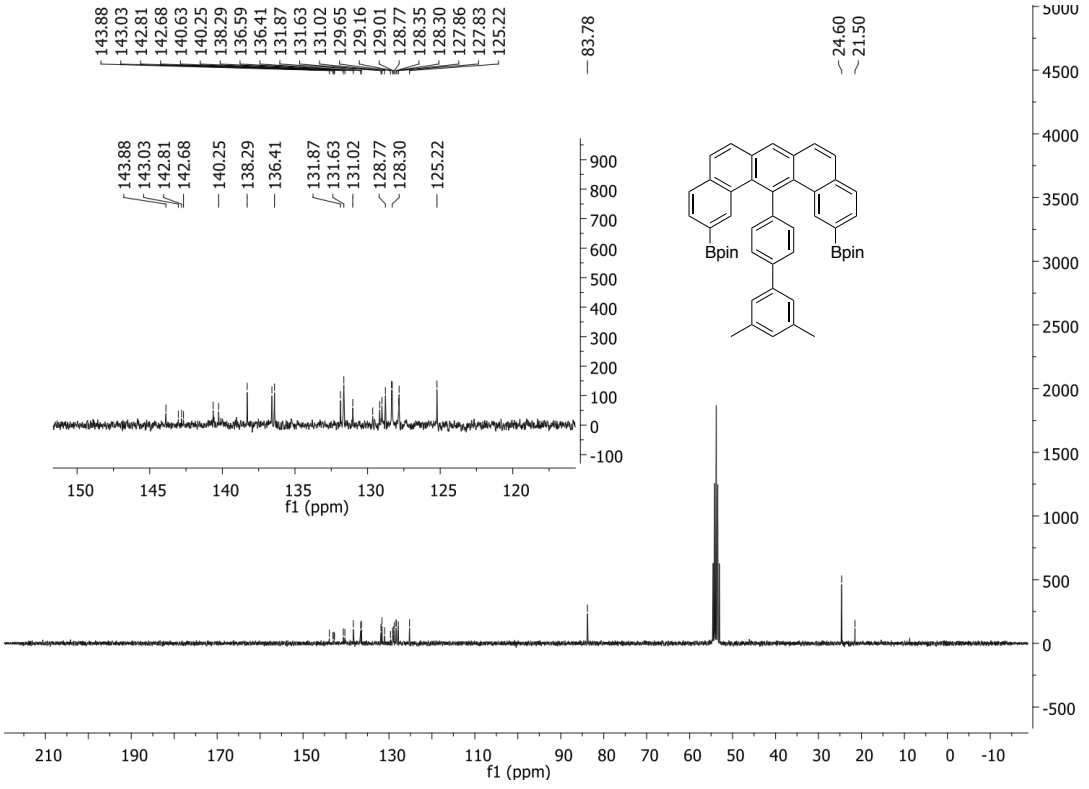
4.01
2.19
2.21
4.16
4.34
0.94
6.14
24.00
f1 (ppm)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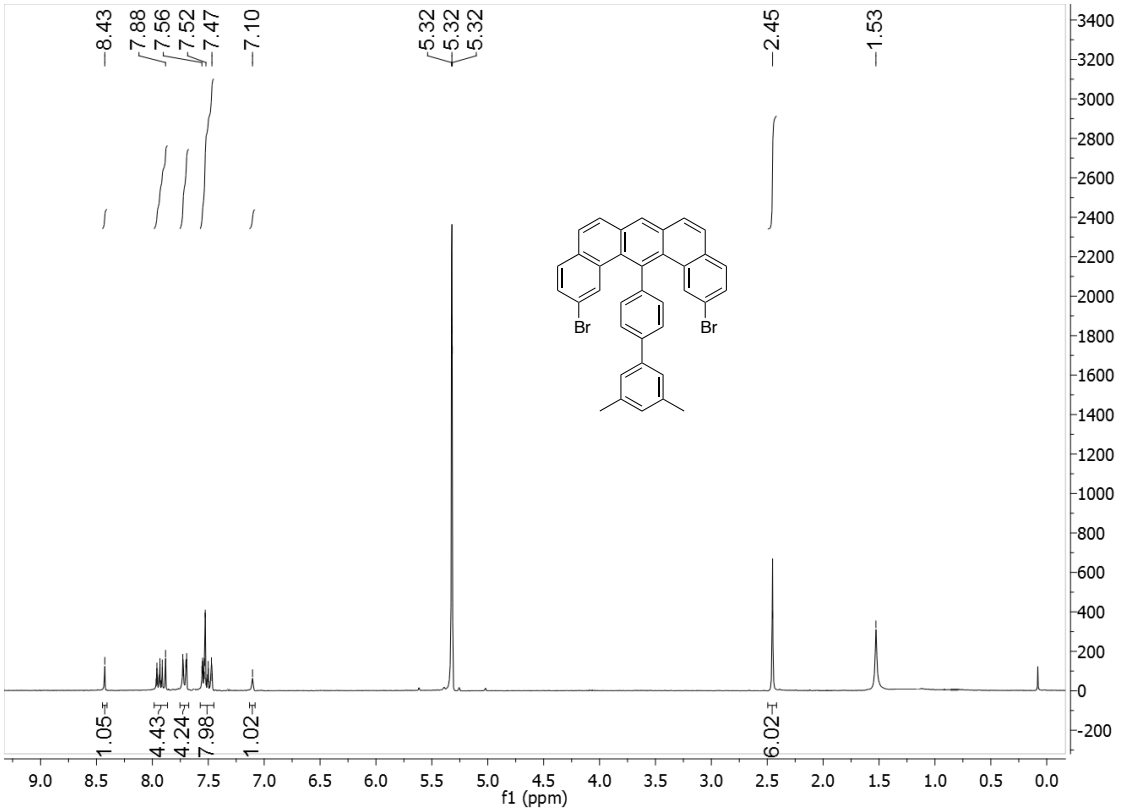
Bpin
Bpin
f1 (ppm)
f1 (ppm)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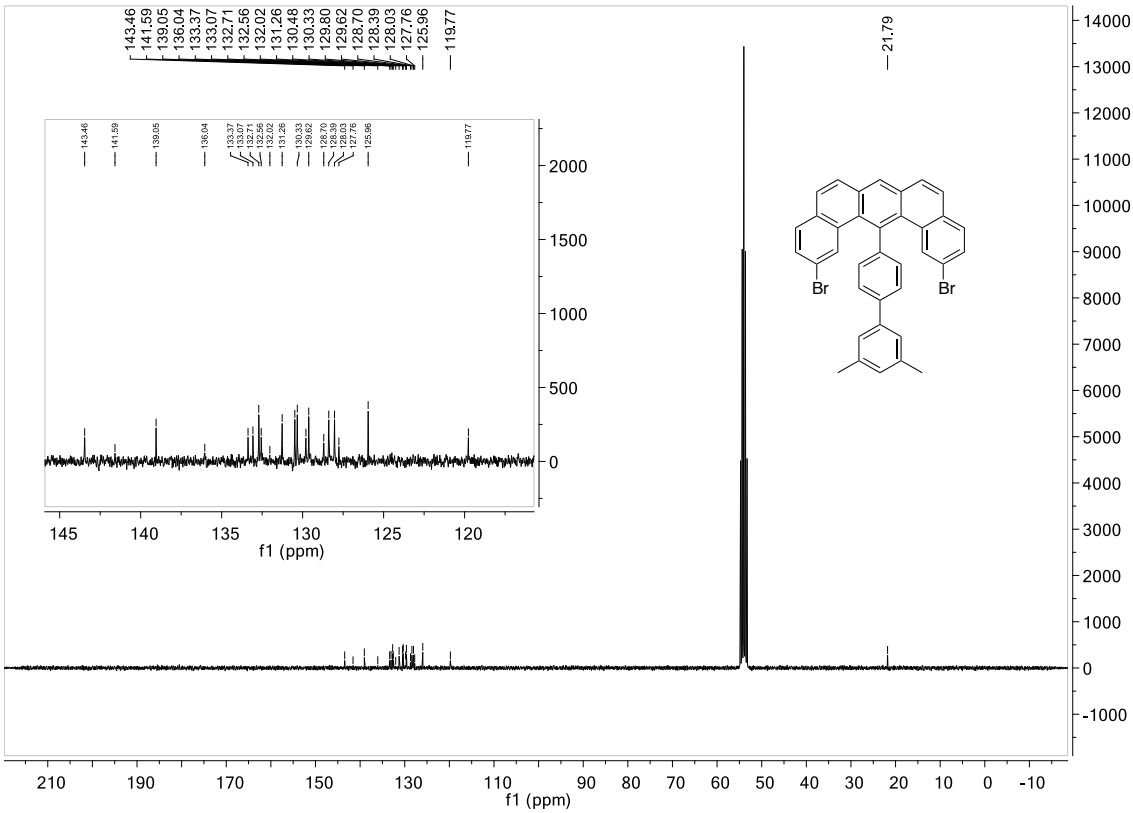
f1 (ppm)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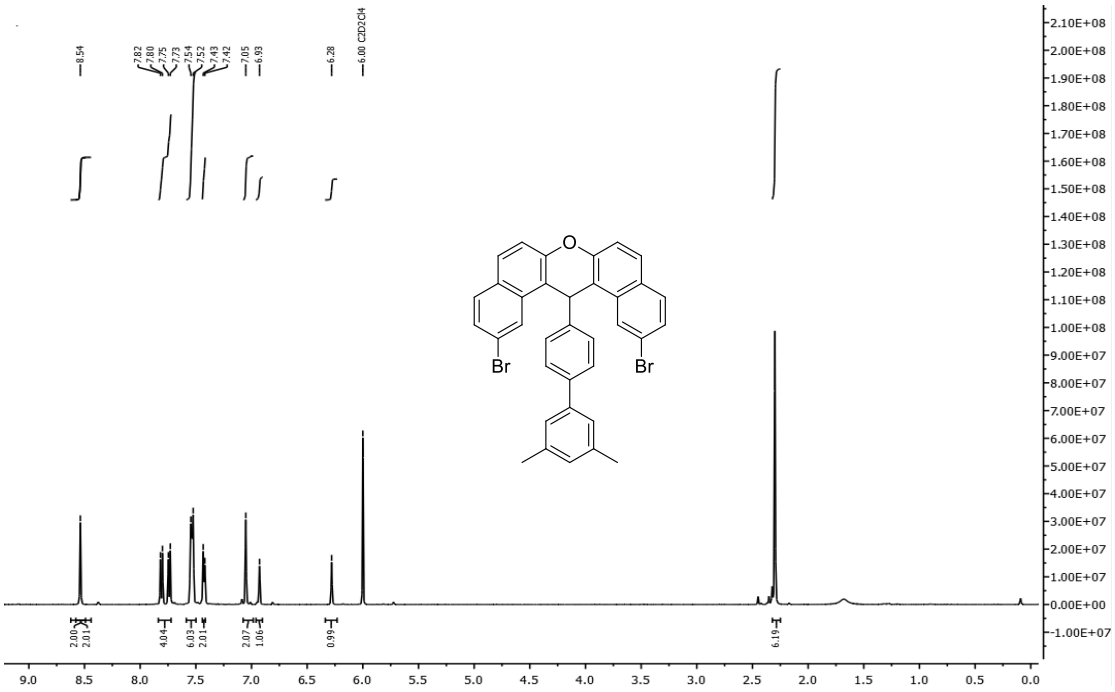
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Br
Br
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2.01
2.07
1.06
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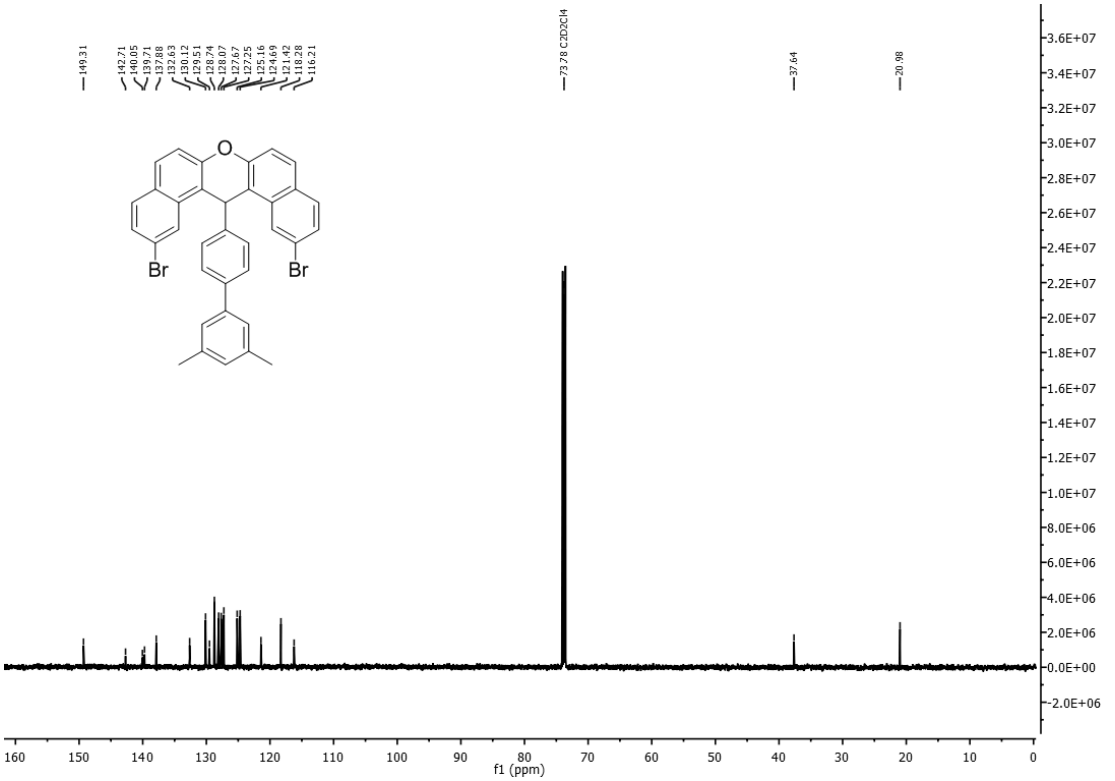
6.19
f1 (ppm)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O
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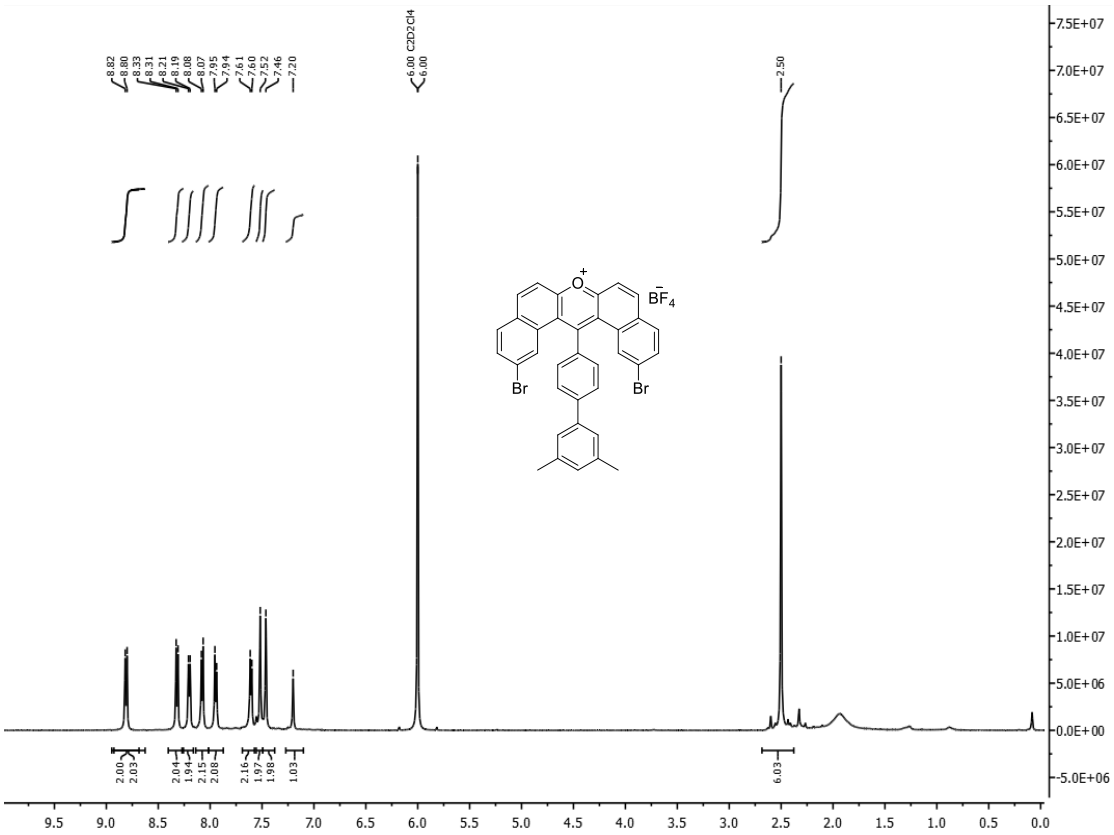
Br
f1 (ppm)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BF4
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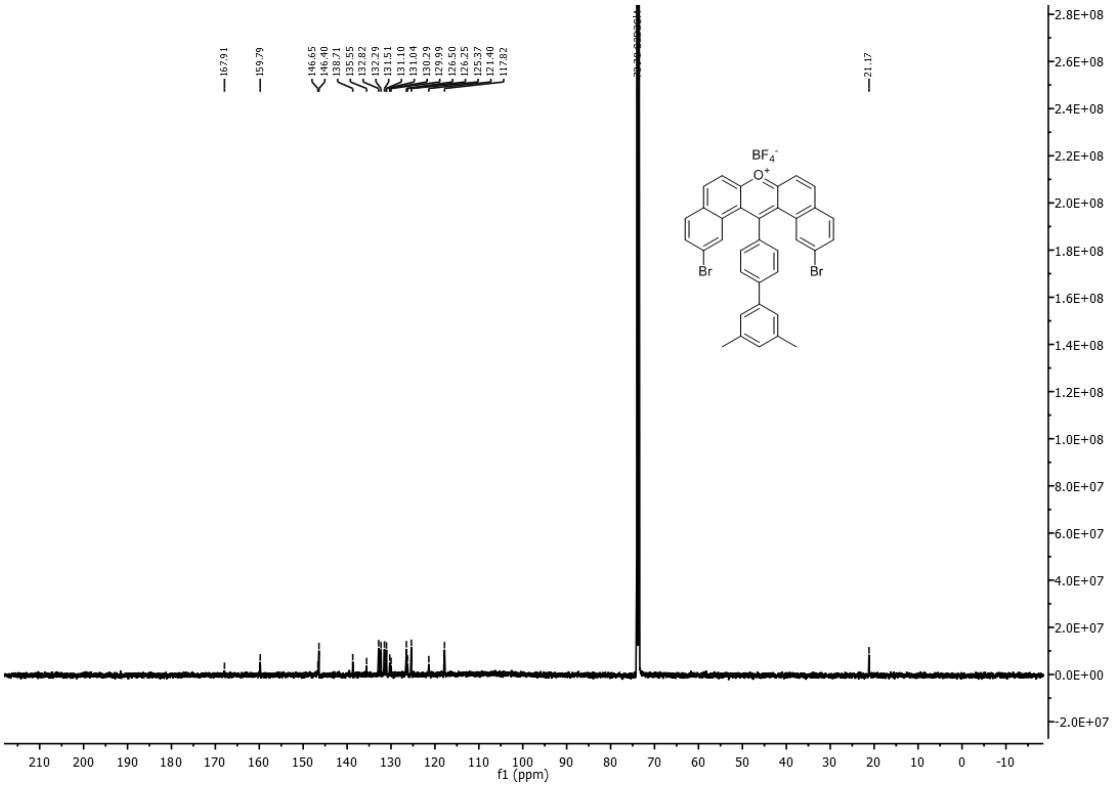
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Br
f1 (ppm)

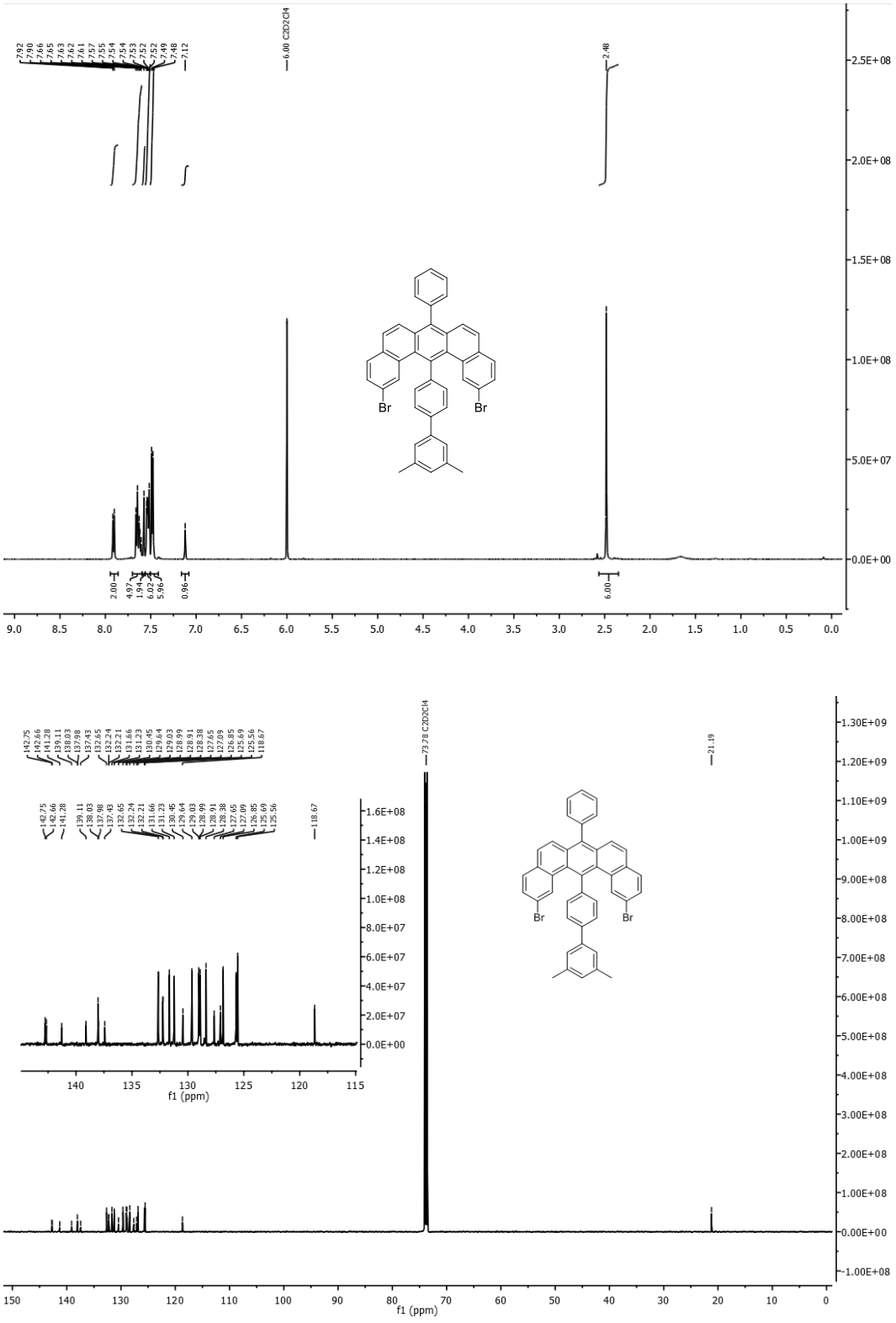

Fig. S8. ^{1}H and ^{13}C NMR spectra. NMR spectra of all the intermediate products and the molecular precursors.

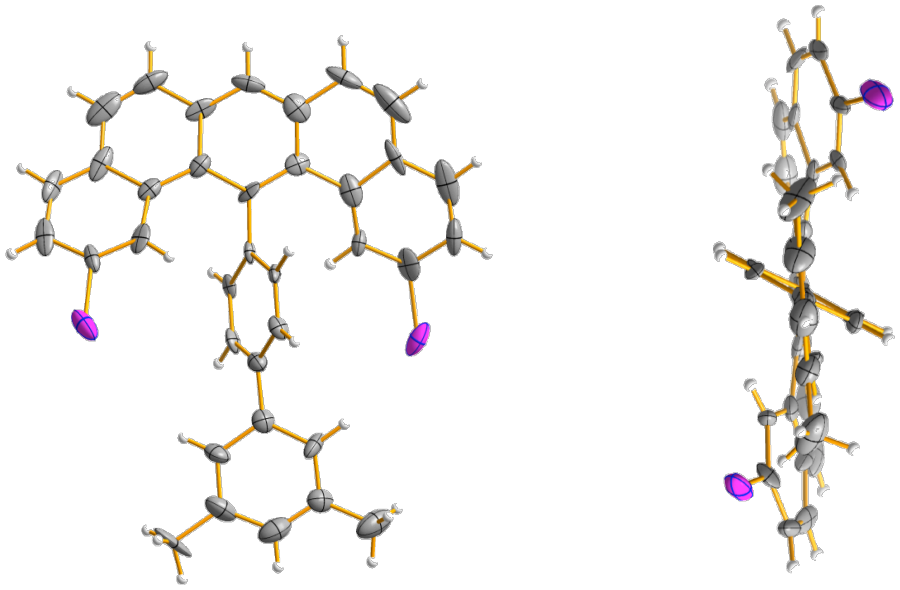

Figure S9. Single crystal structure of monomer **1a** (left: front view; right: side view). See also Supporting File 'single crystal of BrBD-Umbrella.cif'.

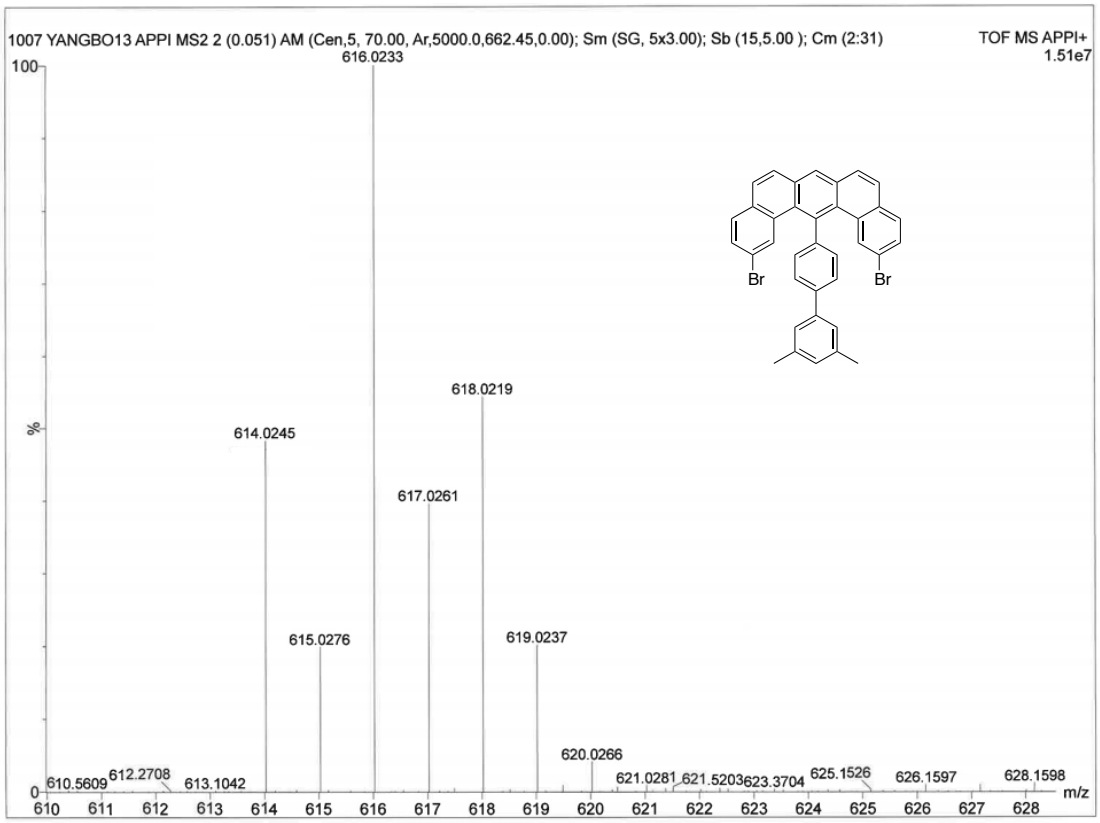

Figure S10. High-resolution APPI-TOF mass spectra of **1a**.

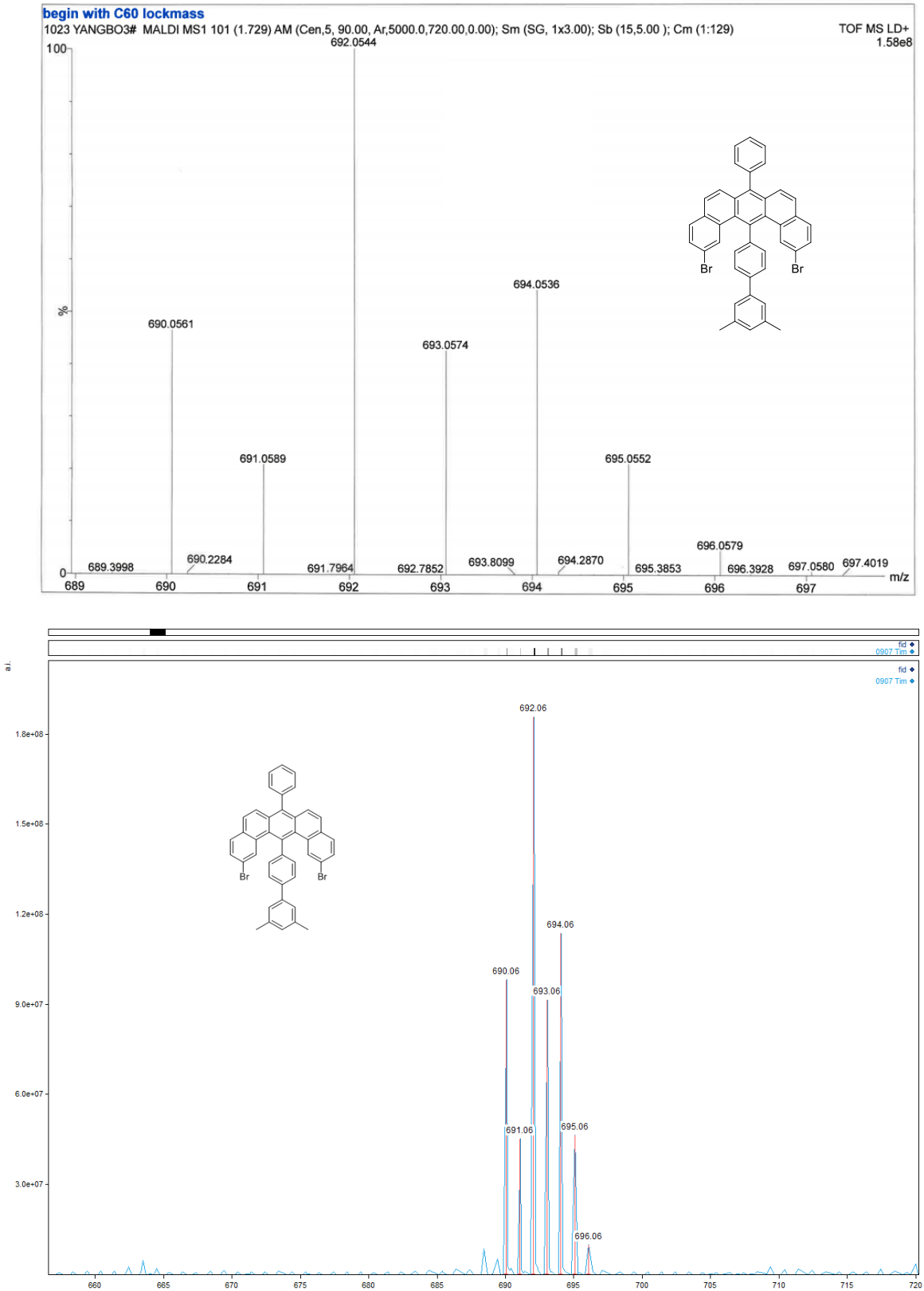

Figure S11. High-resolution MALDI-TOF mass spectra (top) and MALDI-TOF mass spectra (bottom, comparing with estimate mass spectrum (red line)) of **1b**.

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